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MACRO- AND MICROMECHANICAL RESPONSE OF GLASS FIBRE-REINFORCED POLYPROPYLENE TO PIGMENTAL IMPURITIES

The present study investigates the influence of pigmental impurities on glass fibre-reinforced polypropylene using model compounds to simulate the behaviour of recyclate-based compositions. Most industrial-quality (containing recyclate) PP compounds are black coloured (using carbon black pigment), with an almost unavoidable presence of inorganic white pigment (e.g. titanium dioxide) impurities. There are widespread beliefs in the compounding industry that such impurities have a detrimental effect on the mechanical properties of glass fibre-reinforced compounds, but up to now no systematic study of this problem from the industrial point of view has been reported. For this purpose, a range of compounds was prepared on a twin-screw compounding line and the properties were evaluated, with special focus on the mechanical properties. The results confirmed the strong influence of some white pigments, particularly titanium dioxide, and rejected the thesis of the detrimental action of carbon black.

Keywords: polypropylene, recyclate, short glass fibre, titanium dioxide, carbon black, zinc sulphide, zinc oxide

INTRODUCTION

Growing environmental and economic pressure on the plastic industry to close the material loop results in the increased use of recyclates. For applications that are not mechanical demanding, where pure, unmodified recyclates are used, the pigment content (from the source of the recyclate like printed packaging film) poses no significant problem. In the case of technical, modified compounds, such impurities could influence the properties of the final product, resulting in a restricted number of successful applications. Industrial compounding, i.e. the modification of properties due to the use of fillers and reinforcements (among which glass fibres are the most important), has found that using recyclates as a basis for the process strongly influences the mechanical properties, especially in the case of glass fibres, and since this is also one of the most important compounding activities, industry research during the trial-and-error process has found solutions to overcome existing limitations [1-3], but without direct explanation of the occurring discrepancies, sound reasons or scientifically proven measures. The author of this article tried to find the proof and answers, based on model compounds containing definite amounts of the three most important white as well as one black pigment.

MATERIALS AND METHODS

All the compounds were prepared on a pilot-scale twin screw, co-rotating compounding extruder (Nanjing Giant SHJ-36, screw diameter 36 mm, L/D 40) with an under-water pelletizing unit (GALA 5). The processing conditions and temperature settings are summarized in Table 1. The materials used for the preparations of the compounds are presented in Table 2. Table 3 shows the compounds that were produced.

Granulates of the compounds were injection moulded into test specimens (ISO 20753 Type A1 and B1, and discs of diameter 60 mm and thickness 3.7 mm) using a Battenfeld BA 500/200 CD (clamping force 500 kN, screw diameter 35 mm), and processing parameters according to ISO 1873.

TABLE 1. **Processing conditions**

Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Bypass valve	Die-plate	Water	Screw speed	Output
[°C]										[rpm]	[kg/h]
230	235	240	240	240	230	220	230	240	50	300	30

TABLE 2. **Materials used in this study**

Material	Designation in text	Commercial designation	Specification	Supplier
Polymer matrix	PPH	100-CA50	MFI (230°C/2.16 kg) 50 dg/min	INEOS Olefins & Polymers, Köln, Germany
Glass fibre	GF	TGFS 203P	Fibre length: 4.5 mm Filament diameter 10 µm Fibre tensile strength: 2000 MPa	Taiwan Glass Ind. Corp., Taipei, Taiwan
Coupling agent	PP-g-MAH	Bondyram 1001	MAH content ~ 1 wt.%	Polyram Plastic Industries, Ram On, Israel
Titanium dioxide	TiO_2	Ti-Pure R-105	Median particle size: 0.31 µm Density: 4.0 g/cm^3	Chemours, Dordrecht, Netherlands
Zinc oxide	ZnO	Zinkweiss HARZSIEGEL CF	Density: 5.6 g/cm^3 Specific surface area: 5 m^2/g	Norzinco, Goslar, Germany
Zinc sulphide	ZnS	SACHTOLITH HD-S		Venator, Duisburg, Germany
Carbon black (CB) master-batch	CBM	PolyPlast Black FC 7309 PP	Carrier: PP Pigment: 40 wt.% P-Type carbon black	Polyplast Müller, Straelen Germany
Antioxidants package	AO	–	mixture of phenolic and phosphite antioxidants	PCW, Eilenburg, Germany
Processing aids	PA	–	mixture of external and internal waxes	PCW, Eilenburg, Germany

TABLE 3. **Composition of investigated compounds [wt.%]**

Component	PP GF30 NAT	PP GF30 WT1	PP GF30 WT2	PP GF30 WT3	PP GF30 BK1	PP GF30 BK2	PP GF30 BK3	PP GF30 BK4
PPH	67.5	65.5	65.5	65.5	65.75	64.5	62	60.75
GF	30	30	30	30	30	30	30	30
PP-g-MAH	1.5	1.5	1.5	1.5	2	2	2	2
TiO_2	0	2	0	0	0	0	0	0
ZnO	0	0	0	2	0	0	0	0
ZnS	0	0	2	0	0	0	0	0
CBM (CB)[a]	0	0	0	0	1.25 (0.5)	2.5 (1)	5 (2)	6.25 (2.5)
AO	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
PA	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Total	100	100	100	100	100	100	100	100

[a] CB – carbon black content calculated from masterbatch addition level

The tensile properties were tested using a universal tensile testing machine Instron/Kögel FPG 7/20-020 equipped with a 20 kN load cell (Kögel, Leipzig, Germany). According to ISO 527, the test speed of 1 mm/min was used to determine the tensile modulus, and 5 mm/min for tensile strength. The impact strength was measured using a Zwick 5102.100/00 (Zwick, Ulm, Germany) with 4 J pendulum according to ISO 179 (conditions 1eA for notched impact and 1eU for un-notched impact).

The fibre length and orientation distributions were analysed according to ISO 22314 using a VHX6000 light microscope (Keyence, Osaka, Japan). For every compound a small sample taken from the middle of the universal tensile specimen A1 was calcined at 625°C; the obtained ash was dispersed in water with a drop of surfactant and evaluated after being transferred to a microscope glass. The specific properties were calculated from the raw measurement of 300 individual fibres of each compound according to following formulas (1) and (2). The number-average fibre length (L_n) is expressed as follows (1):

$$L_n = \frac{\sum_{i=1}^{n} n_i L_i}{\sum_{i=1}^{n} n_i} \quad (1)$$

where L_i is the length of i-th fibre, n is the number of fibres measured. The weight-average fibre length (L_w) Formula (2):

$$L_w = \frac{\sum_{i=1}^{n} n_i L_i^2}{\sum_{i=1}^{n} n_i L_i} \quad (2)$$

To determine the influence of pigmentation on the tensile properties, the following equations (3)-(9) were used [4-8], starting with the modified rule of mixtures (MROM, 3):

$$\sigma_C = \chi_1 \chi_2 v_F \sigma_F + v_M \sigma_M \quad (3)$$

where σ_C, σ_F and σ_M are respectively the composite, fibre and matrix tensile strength, v_F and v_M are the fibre and matrix volume fractions, χ_1 is the fibre orientation

factor, χ_2 is the fibre length factor. Fibre orientation factor χ_1 was calculated using Equation (4):

$$\chi_1 = 2\langle cos^2\theta\rangle - 1 \quad (4)$$

where $\langle\cos^2\theta\rangle$ is the average value of fibre orientation at angle θ with regard to the flow direction/tensile load, subsequently calculated from measured fibre ellipses in a sample cross-section according to Equation (5):

$$\cos\theta = W/L = 4A/\pi L^2 \quad (5)$$

where W and L are the ellipse minor and major axes, and A is the ellipse area. The determination of $\cos\theta$ usually relies on the right-hand side of Equation (5) since measurement of the minor axis length is the source of a larger error in comparison to measurement of the area. The average fibre orientation factor used in Equation (5) is calculated using Formula (6), from the measured cos θ values (Equation (5)):

$$\langle cos^2\theta\rangle = \frac{\sum_i[N(\theta_i)cos^2\theta_i]}{\sum_i[N(\theta_i)]} \quad (6)$$

where $N(\theta_i)$ is number of fibres measured at angle θ_i.

Fibre length factor χ_2 can be calculated using Equation (7) and (8), respectively:

$$\chi_2 = \frac{L_W}{2L_C} \quad \text{for } L_W < L_C \quad (7)$$

$$\chi_2 = 1 - \frac{L_C}{2L_W} \quad \text{for } L_W \geq L_C \quad (8)$$

where L_C is the critical fibre length. L_c is obtained from fitting Equation (3) to the tensile results.

Using the calculated critical fibre length, the interfacial shear strength (IFSS) between the fibres and matrix, τ, can be calculated using the following equation:

$$\tau = \frac{\sigma_F d_F}{2L_C} \quad (9)$$

where d_F is the fibre diameter, other symbols were explained above.

The glass fibre content was tested according to ISO 3451 (modified method A - rapid ashing at 800°C) in a Phoenix microwave furnace (CEM, Matthews, USA).

The melt flowability was determined with an MFI tester Cflow BMF-003 (Zwick, Ulm, Germany) at 230°C/2.16 kg load according to ISO 1133, connected to an analytical scale AE200S (Mettler-Toledo, Greifensee, Switzerland). The density was measured according to ISO 1183 (buoyancy method, ethanol as the immersion fluid) using an electronic balance AS 82/220 X2 (Radwag, Radom, Poland), equipped with a density attachment.

The colour properties were determined according to DIN 5033 with a Spectraflash 450 X (Datacolor, Lawrenceville, USA) in reflectance mode, geometry d/8 SPIN (specular component included), illumination D65, extended standard observer 10°. From the reflectance spectra, CIE L^*a^*b (1976) colour values were calculated: L^* – lightness, a^* – red-green balance, b^* – yellow-blue balance, additionally colour difference ΔE^* for specific samples. To determine the opacity of white-coloured compounds, two reflectance measurements were made with samples (discs, thickness 3.7 mm) placed over white and black backgrounds, and the YOB/YOW contrast ratio of lightness factors Y (CIE XYZ 1931) was calculated.

RESULTS

White compounds

The results of the tests are summarized in Table 4. As can be clearly seen, colouration with white pigments has a very strong influence on most of the mechanical properties, the most deteriorated one being impact strength, which in the worst case (compound WT1, 2% titanium dioxide), decreased by roughly 40% (see Table 5).

TABLE 4. **Properties of white compounds and reference (mean value and standard deviation)**

Property	PP GF30 WT1	PP GF30 WT2	PP GF30 WT3	PP GF30 NAT
Density [g/cm^3]	1.119 ± 0.001	1.120 ± 0.001	1.119 ± 0.001	1.094 ± 0.001
Ash [%]	32.5 ± 0.1	31.6 ± 0.1	31.6 ± 0.1	29.8 ± 0.1
MFI [dg/min]	22.3 ± 1	14.9 ± 1	21.0 ± 1	16.3 ± 1
Tensile strength [MPa]	73.8 ± 1.3	85.7 ± 0.7	82.1 ± 2.0	95.5 ± 0.3
Elongation at yield [%]	3.6 ± 0.2	3.9 ± 0.2	3.8 ± 0.2	4.4 ± 0.1
Elongation at break [%]	3.7 ± 0.1	3.9 ± 0.2	3.8 ± 0.1	4.4 ± 0.1
Tensile modulus [MPa]	5900 ± 199	5990 ± 374	5950 ± 336	6920 ± 225
Charpy impact strength [kJ/m^2]	32 ± 2	42 ± 1	38 ± 1	54 ± 3
Charpy impact strength notched [kJ/m^2]	5.5 ± 0.3	8.1 ± 0.5	7.1 ± 0.2	9.7 ± 0.3
Colour parameters, CIE L*	92.17	92.12	86.27	–
CIE a*	–0.68	–1.31	–2.26	
CIE b*	3.20	3.22	1.72	
ΔE*	0	0.64[1]	6.291	
Contrast ratio Y_{OB}/Y_{OW} [%]	100	100	98.85	–

[1] in reference to sample coloured with TiO_2 (WT1)

TABLE 5. **Percentage deviation of MFI and mechanical properties in reference to uncoloured compound**

Property	PP GF30 WT1	PP GF30 WT2	PP GF30 WT3
MFI	37	–9	29
Tensile strength	–22	–9	–14
Tensile modulus	–16	–14	–14
Charpy impact strength	–41	–22	–30
Charpy impact strength notched	–43	–16	–27
Average deviation[a]	–30	–16	–21

[a] without MFI

The increase in the melt flow index for compound WT1 (titanium dioxide) and WT3 (zinc oxide) indicates matrix (polypropylene) degradation during processing (+37 and +29% respectively), which correlates well with the deterioration of the mechanical properties of the mentioned compounds. The results of the fibre length and its distribution, as well as micromechanical data (critical fibre length, fibre efficiency factor and interfacial shear strength) are presented in Table 6 and Figures 1 and 2.

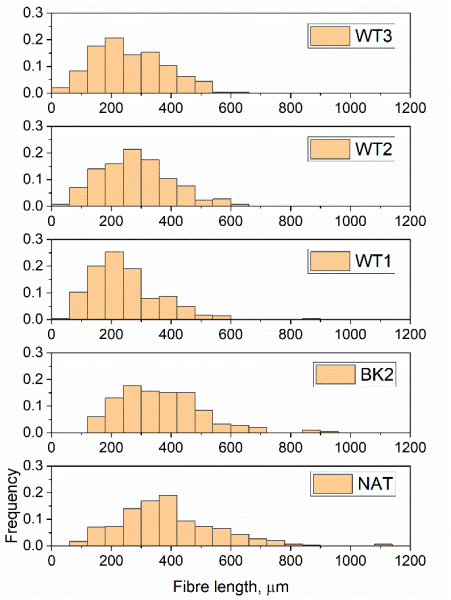

Fig. 1. Fibre length distributions (FLD) of tested compounds and reference

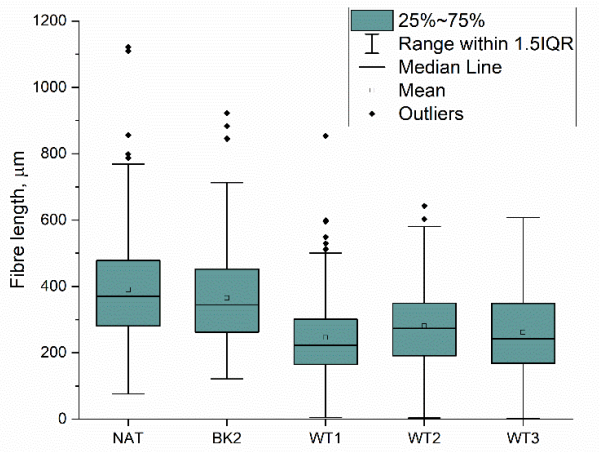

Fig. 2. Numerical presentation of FLD data of tested compounds and reference (IQR – interquartile range)

The above presented results may suggest the use of both ZnS and ZnO as equally performing pigments for the colouration of glass fibre containing compounds. However, the colour properties (CIE L* – Table 4, as well as VIS reflection spectra, Fig. 3) show why zinc oxide is not the best choice when opacity is needed. At almost all wavelengths, the reflection coefficient of the sample containing zinc oxide lies below the titanium white and zinc sulphide spectra, which means zinc white is half-translucent.

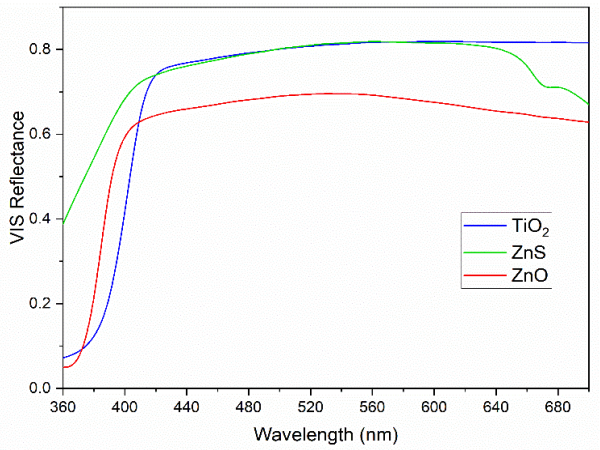

Fig. 3. Reflection spectra of white coloured compounds PP GF30 WT1-3

TABLE 6. **Fibre length and fitted micromechanical results for white compounds and reference**

Property	PP GF30 WT1	PP GF30 WT2	PP GF30 WT3	PP GF30 NAT
Mean fibre length L_n [µm]	246	280	261	390
Weighted mean fibre length L_w [µm]	301	330	315	458
L_w/L_n	1.23	1.18	1.21	1.18
Standard deviation [µm]	117	118	119	164
Critical fibre length L_c [µm]	484	425	423	515
Fibre performance factor $\chi_1\chi_2$	0.1841	0.2296	0.2205	0.2635
IFSS [MPa]	20.7	23.5	23.6	19.4

The reason for this is the significantly lower refractive index of ZnO (nD = 2.02) in comparison to the other pigments (TiO_2 2.76, ZnS 2.37) [9]. The remarkably lower reflection of zinc sulphide starting at 640 nm is caused by the small admixture of cobalt ions, which the manufacturer found best to stabilize the crystal lattice of ZnS against UV-light. Otherwise, the pigment undergoes molecular rearrangement, resulting in a loss of whiteness (greying) [10]. Calculated from the Fresnel formula for external reflection at the mediums border (10), the rough estimation of hiding power HP of the investigated pigments shows that the zinc sulphide content needs to be almost doubled to reach the titanium white level and that of zinc white (zinc oxide) should be four times higher (Table 7).

$$HP = \frac{(n_P - n_M)^2}{(n_P + n_M)^2} \times 100 \qquad (10)$$

where: HP – relative hiding power of white pigment due to scattering, n_P – pigment refractive index, n_M – matrix refractive index (for polypropylene 1.50) [11].

TABLE 7. **Relative hiding power of white pigments**

Pigment	Refractive index	*HP*	*HP* Relative	Pigment demand factor 1/HP Rel.
Titanium dioxide	2.76	8.75	100	1.00
Zinc sulphide	2.37	5.05	57.8	1.73
Zinc oxide	2.02	2.18	24.9	4.00

Black compounds

The results of the tests performed on black compounds are summarized in Table 8.

TABLE 8. **Properties of black compounds (mean value and standard deviation)**

Property	PP GF30 BK1	PP GF30 BK2	PP GF30 BK3	PP GF30 BK4
Density [g/cm^3]	1.104 ± 0.001	1.104 ± 0.001	1.113 ± 0.001	1.121 ± 0.001
Ash [%]	29.3 ± 0.1	29.8 ± 0.1	30.0 ± 0.1	29.4 ± 0.1
MFI [dg/min]	12.1 ± 1	11.3 ± 1	10.2 ± 1	9.66 ± 1
Tensile strength [MPa]	87.9 ± 0.4	91.5 ± 1.4	87.8 ± 0.4	89.9 ± 1.3
Elongation at yield [%]	4.4 ± 0.2	4.4 ± 0.2	4.3 ± 0.1	4.2 ± 0.1
Elongation at break [%]	4.6 ± 0.2	4.4 ± 0.2	4.4 ± 0.1	4.4 ± 0.1
Tensile modulus [MPa]	6280 ± 92	6470 ± 356	6320 ± 385	6400 ± 549
Charpy impact strength [kJ/m^2]	51 ± 1.8	46 ± 2.5	43 ± 2.4	48 ± 2.0
Charpy impact strength notched [kJ/m^2]	8.1 ± 0.4	8.3 ± 0.4	8.4 ± 0.4	8.1 ± 0.2
Colour parameters,				
CIE L*	25.27	25.01	24.95	25.39
CIE a*	–0.03	–0.03	–0.05	–0.03
CIE b*	–0.02	0.01	–0.10	–0.09
ΔE*	-	0.26[a]	0.33[a]	0.14[a]

[a] in reference to sample BK1.

As can be seen, colouration with carbon black (in the form of a masterbatch) has a negligible influence on the tensile properties (largest deviation 10%), impact strength being the most affected (up to –19%); the overall average deviation lies at roughly 10%. It is worth mentioning that the mechanical properties deviate independent of the carbon black content (Tables 8 and 9).

TABLE 9. **Percentage deviation of MFI and mechanical properties of black compounds in reference to uncoloured one**

Property	PP GF30 BK1	PP GF30 BK2	PP GF30 BK3	PP GF30 BK4
MFI	–26	–31	–37	–41
Tensile strength	–7	–4	–7	–5
Tensile modulus	–10	–8	–10	–9
Charpy impact strength	–6	–15	–19	–11
Charpy impact strength notched	–16	–14	–13	–16
Average deviation[1]	–10	–10	–12	–10

[1] without MFI

The carbon black content has the strongest effect on the viscosity of the compounds (measured via MFI). The MFI decreases by up to 41% for compound BK4 (2.5% carbon black) (Table 9). The colour properties are not dependent on the carbon black content either (Table 8).

As there is no influence of the carbon black content on the mechanical properties of the investigated compounds, the fibre length distribution (FLD) and orientation (FOD) were tested only for one compound, namely BK2; the results are presented in Table 10 and Figures 1 and 2.

TABLE 10. **Fibre length and fitted micromechanical results for black compound BK2**

Property	PP GF30 BK2	PP GF30 NAT
Mean fibre length L_n [µm]	365	390
Weighted mean fibre length L_w [µm]	417	458
L_w/L_n	1.14	1.18
Standard deviation [µm]	139	164
Critical fibre length L_c [µm]	498	515
Fibre performance factor $\chi_1\chi_2$	0.2476	0.2635
IFSS [MPa]	20.1	19.4

DISCUSSION

The suggestion that pigments influence the mechanical properties of glass fibre-reinforced thermoplastic compounds due to milling action could easily be proved using the fibre length data presented in Tables 6 and 10. For any of the investigated compounds, both the number as well as weight average fibre length are obviously lower than for an uncoloured one, also the

FLD width is narrower (Figs. 1 and 2). In addition, the L_w/L_n ratio, as stipulated in norm ISO 22314, does not contain additional information since all the calculated values lie in the range of the uncoloured compound (1.17-1.22). The calculated micromechanical parameters (critical fibre length and interfacial shear strength) lie at the expected level [7] for fully coupled PP GF; the same is valid for the fibre performance factor, which correlates well with the fibre length distribution.

The results presented in Table 6 confirm the strong influence of titanium white (WT1) on the mechanical properties of the compound. Compared to the reference compound, both the mean and weighted fibre length, as well as the fibre performance factor, are significantly lower, indicating significant degradation of the fibres during processing. For the two other white pigments, zinc oxide and zinc sulphide, the fibre length and performance factor correlate well with the mechanical properties, in slight favour of the latter. Comparing the results of the mechanical and colour properties, the obvious winner is zinc sulphide, exhibiting the lowest deterioration of properties and good performance as a white pigment (hiding power and colour purity).

Evaluation of the results for the black compounds (Tables 8-10) reveal that a slight weakening of the mechanical performance, independent of carbon black content, is caused by increased shear forces in the melt induced by carbon black. The fibre length distribution (Figures 1 and 2, compound BK2) looks very similar to the FLD of the uncoloured compound NAT, except for some flattening of the fibre frequency, so not a pigment property, but increased viscosity is the reason for this aberration. Just 0.5% of carbon black (sample BK1) causes a 26% decrease in the measured flowability and thus increased the forces acting on the fibres during processing.

Based on the tribological approach for two-body abrasive wear [12], wear factor W (in the present case the milling of glass fibres due to pigment action) depends on the hardness ratio of abrasive H_A (pigment) to material H_M (glass-fibre), as in Formula (11) [13]:

$$W \approx \frac{H_A}{H_M} \qquad (11)$$

Calculating above mentioned hardness ratio for every pigment and plotting the relative loss in the mechanical properties as a function of H_A/H_M reveals undoubtedly the same pattern for each property loss (Fig. 4), the impact properties being the most deteriorated. At the hardness ratio of 0.07 (carbon black), the property loss reaches roughly 15% for the impact strength. In the range of 0.23-0.27 (ZnS and ZnO) the impact strength loses a further 15%, and finally, at the hardness ratio of 0.9 (TiO_2) the ultimate loss in impact (40%) and tensile strength (23%) is observed. The latter value of the hardness ratio coincides well with the data indicated by Nosal [13] for the threshold of change from a mild to abrasive mode of action (0.7-1.1).

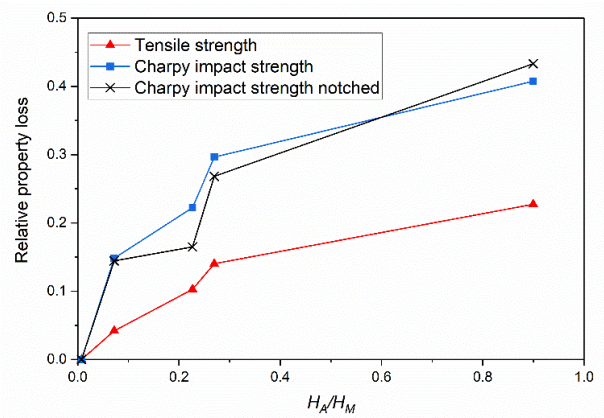

Fig. 4. Relative loss of mechanical properties of PP GF30 compounds in relation to hardness ratio of abrasive (pigment) to glass fibre (material), H_A/H_M

CONCLUSIONS

The relatively simple process of colouring glass fibre-reinforced polypropylene compounds reveals, after careful investigation, a whole spectrum of side effects, undoubtedly influencing the final properties of the product. On the basis of the results presented in this article, the following final conclusions can be formulated:

1. Glass fibres suffer from the presence of pigments during processing due to the milling action of the pigments. The loss in properties scales well with the change in the hardness ratio of the pigment to glass.
2. The only right choice for white pigment is zinc sulphide, which has the least influence on the properties due to its low hardness, but unfortunately in recyclates titanium dioxide is the most common, which leads to a great extent to deterioration of the mechanical properties of the compounds, limiting the spectrum of possible, demanding applications.
3. Any more abrasive forms of carbon black (like carbon black HAF types, carbon fibres) will lead to similar property losses as in the case of mildly abrasive white pigments like zinc oxide.
4. The exact mode of action of pigments on glass fibres should be further investigated using model substances with other hardness ratios, e.g. lower than 0.25, as well as in the range 0.25-0.9 and above.

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