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Influence of the Composition of Polypropylene/Organoclay Nanocomposite Fibers on their Tensile Strength

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The statistical method "Experimental Design" was applied to optimization of weight composition of isotactic poly(propylene)/organoclay (iPP/org.clay) nanocomposite fibers from the standpoint of achieving the desired tensile strength at break as one of the significant mechanical properties. These properties were studied on fibers prepared from samples of iPP/org.clay nanocomposites of differing compositions. According to the statistical program, there were thirteen samples prepared containing the organoclay filler,NANOFIL, in the concentration range 0.5 to 4.9 wt%, and compatibilizer, an iPP grafted with maleic anhydride (iPP-g-MA) of concentration from 1 to 5 wt%. The samples were spun, and the obtained fibers underwent measurements of tensile strength at break, σ. Evaluation of the obtained data, led to the establishment of an optimal compatibilizer to filler ratio (Comp./Fill.) of 0.16 to 2.76, for which the tensile strength is higher than for unfilled iPP fibers.

Keywords polypropylene nanocomposite, organophilic layered silicate filler, polypropylene grafted with maleic anhydride-compatibilizer, polypropylene nanocomposite fibers

Introduction

In recent years, a number of papers have dealt with the influence of individual components on the properties of isotactic polypropylene (iPP) nanocomposites (1–10). The effect of filler and compatibilizer on morphology and physico-mechanical properties can influence the commercial potential of this plastic material (8, 9). The iPP nanocomposites were also studied in their fiber form, which in particular, the high orientation of both the iPP matrix and also orientation of filler particles-mostly prepared from layered silicate

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montmorillonite-during the spinning process influenced the mechanical properties of the fibers (2). Fibers prepared from iPP nanocomposites without the presence of the compatibilizer (usually grafted maleic anhydride onto iPP) showed lower tensile strength over the entire investigated concentration range of filler (from 1 to 5 wt%) compared to nanocomposites containing a compatibilizer (2). On the other hand, enhancement of the tensile strength of the fibers brought on by the compatibilizer usually passes a maximum, after which the tensile strength starts to decrease. The decrease of tensile strength is caused by the fact that the compatibilizer, with its lower molecular weight and usually, also with a lower content of crystallinity in comparison with the original iPP matrix, lowers the mechanical properties of the fibers (2).

In this paper, we describe a method called "Experimental Design", which is used for optimization of the weight proportion of the various components in the polymer systems from the standpoint of achieving the desired mechanical properties. We have attempted to approach the interval of the ratio of compatibilizer to filler at which the highest tensile strength can be reached. The measurements of tensile strength of the fibers also provides information about the influence of the proportions of iPP additives, i.e., compatibilizer and filler, on adhesion of nano-particles to the iPP matrix, which is directly proportional to the reinforcing effect of the nano-filler (11).

Experimental

Materials

Isotactic polypropylene-iPP of the type PP-HPF, product of Slovnaft a.s., Bratislava, Slovakia, unstabilized, with MFI (230°C) = 28.7 g/10 min was used as a polymer matrix for preparation of iPP/clay nanocomposites.

The filler, NANOFIL 15, kindly provided by Sued-Chemie AG Muenchen, Germany, is a modified product of montmorillonite, where distearyl-dimethyl-ammonium-chloride chains form the interlayer, was used. NANOFIL is a powder with an average particle size of 25 mm. After dispersion of the particles, the average size of the particles is ~500 nm.

The compatibilizer used was iPP grafted with maleic anhydride (iPP-g-MA) ($M_w = 11\,890\ g \cdot mol^{-1}$), containing 3.5 wt% MA units, and is a commercial product, AR504, (compliments of fa Clariant, German).

During compounding of the composite components, a synergistic stabilization mixture of thermo-oxidation stabilizers, Irganox 1010-**Ix** (pentaerythrityl-tetrakis(3-(3′,5′-di-tert.-butyl-hydroxyphenyl-propionate)) and Irgafos 168-**Is** (2,4-di-tert.butylphenyl)phospite), (both products of fa Ciba) a in ratio Ix/Is = 4 : 1, was added the total.

The amount of stabilizers in iPP nanocomposite was 0.3 wt% related to the amount of iPP.

Proposal for the Optimalization of the Composition of the iPP-NANOFIL-iPP-g-MA Mixture by Method "Experimental Design"

The method "Experimental Design" is used for the arrangement of experiments in order to obtain the most effective mathematical-statistical evaluation. It provides information about the investigated system, which enables one to adjust the composition parameters in order to obtain the required values of the outlet variables.

From the point of view of the data processing of the experimental design, equations are obtained, which can be used for following optimization of the composition of the investigated system (11).

In this paper, a scheme for five levels and two factor experiments was used. The influence of the content of the two additives used in the preparation of iPP nanocomposite was investigated: i.e., the content of filler NANOFIL (Fill), and the content of the compatibilizer (which was iPP-g-MA) of commercial production AR504 (Comp) in the iPP matrix. The content of the individual components in the iPP nanocomposite was decided on the basis of the knowledge of the initial range of 0.5–4.9 wt% NANOFIL and 0.8–5.9 wt% AR504.

From the mathematical-statistical assumptions of the method "Experimental Design" the following independent variables were set:

$$x_1 = \text{Comp/Fill} \quad x_2 = (\text{Comp} + \text{Fill})/\text{iPP}$$

Factor x_1 is the weight ratio of compatibilizer to filler.
Factor x_2 is the weight ratio of the whole content of additives to isotactic polypropylene.

Tables 1 and 2 give the conditions and specification of the "Experimental Design".

Preparation of Poly(propylene) Nanocomposite Samples

The iPP powder of about 300 g was melt-blended at 250°C with iPP-g-MA (from 1 to 5 wt%), filler NANOFIL (0.5 to 4.9 wt%) and a mixture of stabilizers, Irganox 1010 and Irgafos 168, in a ratio 4 : 1 of 0.3 wt% in a twin-screw extruder of type W + P ZSK-28, having a screw diameter of 28 mm and length of 112 cm. The nanocomposite was extruded through a 4 × 5 mm outlet and cut into about 10 mm long granules.

Spinning of the iPP Nanocomposites

The granules of iPP nanocomposite were first vacuum dried 3 h at 80°C, and then spun at the rate of $150\,\text{m} \cdot \text{min}^{-1}$. The other parameters of the spinning were as follows: the diameter of the screw die was f = 16 mm, the nozzle contained 13 outlets having a diameter of 0.5 mm at 250°C.

Polymeric strands of iPP nanocomposite issued from the nozzle were wound onto a bobbin. At a temperature of 110°C, the fibers were drawn at a drawing ratio λ of 2, 3, and 4. Before making tensile strength measurements, the fibers were dried for 30 min. at 120°C.

Table 1
Conditions of "Experimental Design"—transformation of coded levels of factors on to their real values

Symbol	Factor	−1.414	−1	0	1	1.414	Step
x_1	Comp/Fill	0.1682	1.7000	5.4000	9.1000	10.6318	3.7
x_2	(Comp + Fill)/iPP	0.046	0.050	0.060	0.070	0.074	0.01

Table 2
Conditions for individual trials for preparation of iPP fibers

Sample No.	Coding levels of factors		Real levels of factors		Composition of iPP nanocomposite fibers (wt%)		
	x_1	x_2	x_1	x_2	iPP	NANOFIL	iPP-g-MA AR504
1	−1	−1	1.70	0.05	95.24	1.76	3.00
2	1	−1	9.10	0.05	95.24	0.47	4.29
3	−1	1	1.70	0.07	93.46	2.42	4.12
4	1	1	9.10	0.07	93.46	0.65	5.89
5	−1.414	0	0.17	0.06	94.34	4.85	0.81
6	1.414	0	10.63	0.06	94.34	0.49	5.17
7	0	−1.414	5.40	0.05	95.62	0.69	3.70
8	0	1.414	5.40	0.07	93.10	1.08	5.82
9	0	0	5.40	0.06	94.34	0.88	4.78
10	0	0	5.40	0.06	94.34	0.88	4.78
11	0	0	5.40	0.06	94.34	0.88	4.78
12	0	0	5.40	0.06	94.34	0.88	4.78
13	0	0	5.40	0.06	94.34	0.88	4.78

Tensile Strength Measurements

The tensile strength of iPP nanocomposite fibers was calculated from the stress at breakage of the fibers (in cN) as measured on an INSTRON 1112 instrument.

The fiber fineness (long density of the fibers) was determined as an average value from measurements of 10 samples of fibers, 10 m long. The values of the fibers strength given in Table 3 represent an average of 30 measurements of the fibers prepared from the same sample.

The conditions of the measurements: gauge length = 10 cm and deformation rate $v_{ps} = 500\,\text{mm} \cdot \text{min}^{-1}$ were the same for all measurements of samples with different drawing ratios.

Proposal for the Optimization of the Composition of the iPP-NANOFIL-iPP-g-MA Mixture by Method "Experimental Design"

The method "Experimental Design" is used for the arrangement of experiments in order to obtain the most effective mathematical-statistical evaluation. It provides information about the investigated system, which enables one to adjust the composition parameters in order to obtain the required values of the outlet variables.

From the point of view of the data processing of the experimental plan, equations are obtained, which can be used for following optimization of the composition of the investigated system (11).

In this paper, a scheme for five levels and two factor experiments was used. The influence of the content of the two additives used in the preparation of iPP nanocomposite was investigated: i.e., the content of filler NANOFIL (Fill), and the content of the

Table 3
Tensile strength at break $-\sigma_b$ of iPP nanocomposite fibers with various drawing ratios

Sample No.	iPP (wt%)	iPP-g-MA AR504 (wt%)	NANOFIL (wt%)	σ_b (cN/dtex)		
				$\lambda = 2$	$\lambda = 3$	$\lambda = 4$
1	95.24	3.00	1.76	1.12	2.13	3.63
2	95.24	4.29	0.47	1.11	2.18	3.66
3	93.46	4.12	2.42	1.15	2.15	3.21
4	93.46	5.89	0.65	1.22	2.28	3.60
5	94.34	0.81	4.85	0.93	1.62	2.43
6	94.34	5.17	0.49	1.17	2.08	3.51
7	95.62	3.70	0.69	1.28	2.18	3.36
8	93.10	5.82	1.08	1.43	2.11	3.47
9	94.34	4.78	0.88	1.34	2.08	3.57
10	94.34	4.78	0.88	1.09	2.06	3.60
11	94.34	4.78	0.88	1.19	2.17	3.65
12	94.34	4.78	0.88	1.26	2.11	3.75
13	94.34	4.78	0.88	1.20	2.17	3.79

compatibilizer (which was iPP-g-MA) of commercial production AR504 (Comp) in the iPP matrix. The content of individual components in the iPP nanocomposite was decided on the basis of the knowledge of the initial range of 0.5–4.9 wt% NANOFIL and 0.8–5.9 wt% AR504.

From the mathematical-statistical assumptions of the method "Experimental Design" the following independent variables were set:

$$x_1 = \text{Comp/Fill}\quad x_2 = (\text{Comp} + \text{Fill})/\text{iPP}$$

Factor x_1 is the weight ratio of compatibilizer to filler.
Factor x_2 is the weight ratio of the whole content of additives to isotactic polypropylene.

Tables 1 and 2 give the conditions and specification of the "Experimental Design".

Results and Discussion

The samples of prepared iPP nanocomposite fibers were subjected to tensile strength measurement with regard to a dependency on specified factors. Also, the influence of drawing ratio of the fibers on their tensile strength was evaluated (Table 3).

The obtained values from the tensile strength measurements were processed on the basis of the theory of experimental design and utilizing regression analysis of variance by which statistical tests were realized at a 95% level of reliability ($a = 0.05$). The computer program STATIS (1) was used for the evaluation.

The following parameters for the regression model were obtained from computation of measured data using ANOVA and regression analysis methods:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{12}x_1x_2 + b_{11}x_1^2 + b_{22}x_2^2,$$

where

Y—tensile strength of nano-fibers with higher orientation
x_1, x_2—factors in coded co-ordinates
b_0, . . .—regression coefficients

The following symbols will be used during evaluation of experiment:

b_{c0}, . . .—critical values of regression coefficients at probability level 95% (a = 0.05).
S_1—variability described by the linear part of regression equation.
S_2—variability described by thenon-linear part of regression equation.
S_R—residual part of variability.
S_E—experimental variability (experimental error).
S_{LF}—lack of fit variability.
s_{LF}—lack of fit mean square error.
s_E—experimental mean square error.
F_{crit}—critical value of F criteria at a = 0.05.

Results from the evaluation of the "Experimental Design" method indicate that the influence of the investigated factors is not evident at a low drawing ratio $\lambda = 2$ (Tables 4 and 5) in the given experimental region. Neither value from the criteria F_1 and F_2 exceeds the critical value and, neither of the regressive coefficients was statistically significant at a drawing ratio $\lambda = 2$. The changes of the tensile strength in the framework of the experiment were not distinguishable from the experimental error, and because of this they can be considered as values originating from one basic set of data. From this, it follows that for drawing ratio $\lambda = 2$, we are able to determine only the average value of the tensile strength of 1.19 cN/dtex with a standard of the mean square error value 0.034. At higher drawing ratios, $\lambda = 3$ and $\lambda = 4$, the influence of the investigated factors on tensile strength of the fibers was more distinct.

The regression coefficients of the equations, which are statistically important, are printed in bold type.

Fiber tensile strength values resulting from evaluation of the influence of proportion of compatibilizer to filler (Comp/Fill = factor x_1) and the ratio of total content of filler and compatibilizer ((Comp + Fill)/iPP = factor x_2) are presented in Tables 6 to 9.

The response surface for tensile strength dependence on both investigated factors for drawing ratios $\lambda = 3$ and $\lambda = 4$, calculated according to the regression equation are

Table 4
Analysis of variance of factor values drawing ratio $\lambda = 2$

	$\lambda = 2$				
	S_I	f_I	s^2	F	F_{crit}
S_1	0.035	2	0.018	2.08	6.94
S_2	0.099	3	0.033	3.89	6.59
S_R	0.050	7	0.0072	0.84	6.09
S_E	0.034	4	0.0085	1	
S_{LF}	0.016	3	0.0053	0.62	6.59

Table 5
Values of regression coefficients, their critical values and mean square errors for $\lambda = 2$

$\lambda = 2$			
$b_0 = 1.22$	$b_{c0} = 0.115$	$s_{b0} = +/-0.041$	
$b_1 = 0.050$	$b_{ci} = 0.091$	$s_{bi} = +/-0.033$	$s_{LF} = +/-0.073$
$b_2 = 0.044$			$s_E = +/-0.092$
$b_{11} = -0.096$	$b_{cii} = 0.097$	$s_{bii} = +/-0.035$	
$b_{12} = 0.02$	$bc_{ij} = 0.128$	$s_{bij} = +/-0.046$	
$b_{22} = 0.56$			

presented in Figures 1 and 2. From the obtained results, it is evident that the response plane for tensile strength at the higher drawing ratio ($\lambda = 4$) is shifted to the higher values in the experimental area, in comparison with the response surface values for drawing ratio $\lambda = 3$, as would be expected. In both cases, x_1 is the determining factor of the resulting tensile strength of the fibers, i.e., ratio Comp/Fill. Only at lower drawing ratio, $\lambda = 3$, is the factor x_2 also expressed only moderately as a quadratic contribution, producing an insignificant dip in the response surface according to x_2.

The tensile strength of the fibers is shifted more clearly to the higher values upon increasing the ratio Comp/Filler at both drawing ratios ($\lambda = 3$ and $\lambda = 4$), resulting in quadratic bends to the maximum.

A drawing ratio $\lambda = 3$ has a maximum outside of the experimental region and is shifted to the higher values of Comp/Fill. At drawing ratio $\lambda = 4$, it is seen that the top of the maximum is situated at the edge of the experimental area, and again in the region of the high ratio of Comp/Fill. The response surface marked with a broken line in Figures 1 and 2, correspond to the tensile strength of the unfilled, unmodified iPP fiber. The modified fibers, namely at $\lambda = 4$, and containing proper composition of additives, can achieve remarkably higher tensile strength than unmodified.

This evaluation further shows, that the influence of the compatibilizer content, relative to the filler, enhanced with drawing ratio of the fibers (Figures 1 and 2). As far as the important regression coefficients for drawing ratio, $\lambda = 3$, the achieved values

Table 6
Analysis of variance of factor values drawing ratio $\lambda = 3$

	$\lambda = 3$				
	S_I	f_I	s^2	F	F_{crit}
S_1	0.086	2	0.043	**16.78**	6.94
S_2	0.091	3	0.030	**11.78**	6.59
S_R	0.11	7	0.016	6.35	6.09
S_E	0.010	4	0.0026	1	
S_{LF}	0.10	3	0.035	**13.49**	6.59

Note: Values typed in bold are statistically significant.

Table 7
Values of regression coefficients, their critical values and mean square errors for $\lambda = 3$

$\lambda = 3$			
$\mathbf{b_0 = 2.12}$	$b_{c0} = 0.063$	$s_{b0} = +/-0.023$	
$b_1 = 0.10$	$b_{ci} = 0.050$	$s_{bi} = +/-0.018$	$s_{LF} = +/-0.19$
$b_2 = 0.0026$			$s_E = +/-0.050$
$b_{11} = -0.087$	$b_{cii} = 0.053$	$s_{bii} = +/-0.019$	
$b_{12} = 0.02$	$b_{cij} = 0.070$	$s_{bij} = +/-0.025$	
$b_{22} = 0.060$			

Note: Values typed in bold are statistically significant.

were $b_1 = 0.10$ and $b_{11} = -0.087$, whereas for $\lambda = 4$, these values were two times higher. This means that the same change of Comp/Fill ratio produces a two-fold higher increase of tensile strength of the fibers, if the fibers are drawn at $\lambda = 4$ in comparison with fibers obtained at $\lambda = 3$ (Tables 7 and 9). We assume, that at higher drawing ratio of fibers, a higher orientation of the polymer in the fibers is achieved and also the crystallinity increases. So there are formed closely organized structures, resulting in better adhesion at the iPP matrix-filler interface.

From the evaluation of the method "Experimental Design" it is evident that the resulting tensile strength of the iPP fibers modified with the filler NANOFIL and compatibilizer, iPP-g-MA in the investigated experimental area, is influenced mainly by the ratio of Comp/Fill at higher drawing ratios of the fibers (Figure 3).

The results of the statistical evaluation of the "Experimental Design" refer to a very wide concentration interval of the investigated components of the nanocomposite. And so it is not possible to distinguish some effects from experimental error or with greater probability from regression error. Therefore, in the following experiment, we have tried to seek mutual coherence between concentration of the compatibilizer and filler in iPP and their influence on tensile strength of the fibers. We have used the results received from the "Experimental Design", analysis, which has shown that an increase of the fiber tensile strength can be expected in the region of higher ratios of Comp/Fill. There were prepared nanocomposites, which contained the combined concentration Comp + Fill = 5.66 wt%, at which point the ratio Com/Fill varied from 0.34 to 1.5 (sample 14–17). Included in

Table 8
Analysis of variance of factor values drawing ratio $\lambda = 4$

	$\lambda = 4$				
	S_I	f_I	s^2	F	F_{crit}
S_1	0.49	2	0.24	**27.00**	6.94
S_2	0.53	3	0.18	**19.67**	6.59
S_R	0.46	7	0.066	7.30	6.09
S_E	0.036	4	0.0090	1	
S_{LF}	0.43	3	0.14	**15.71**	6.59

Note: Values typed in bold are statistically significant.

Table 9
Values of regression coefficients, their critical values and mean square errors for $\lambda = 4$

$\lambda = 4$			
$b_0 = 3.67$	$b_{c0} = 0.12$	$s_{b0} = +/-0.042$	
$b_1 = 0.24$	$b_{ci} = 0.093$	$s_{bi} = +/-0.034$	$s_{LF} = +/-0.38$
$b_2 = -0.041$			$s_E = +/-0.095$
$b_{11} = -0.27$	$b_{cii} = 0.10$	$s_{bii} = +/- 0.036$	
$b_{12} = 0.09$	$b_{cij} = 0.13$	$s_{bij} = +/-0.047$	
$b_{22} = -0.045$			

this series of samples is sample 5, from "Experimental Design", in which the entire content of additives (Comp + Fill) was 5.66 wt%, but the sample had a very low content of compatibilizer and high content of filler.

The tensile strength of the iPP nanocomposite fibers was compared with the unfilled iPP fibers prepared by the same technique. The composition and tensile strength of the fibers for individual drawing ratios are presented in Table 10. The dependence of the tensile strength of fibers for drawing ratio 2, 3 and 4 on the ratio Comp/Fill without regard to filler content is shown on Figure 3. The values of tensile strength of unfilled iPP fibers for those prepared at corresponding draw ratios are marked with dashed lines. For sample 5, containing a high amount of filler and a low amount of compatibilizer, a decrease of tensile strength of the fibers was observed in comparison with unfilled fiber at all drawing ratios. The difference between filled and unfilled fibers increases with the drawing ratio. When the Comp/Fill ratio was increased from 0.16 to 2.76, there was a relatively sharp increase of tensile strength of fibers observed over the value of unmodified fibers (at a different drawing ratio). Upon further increase of Comp/Fill, the tensile

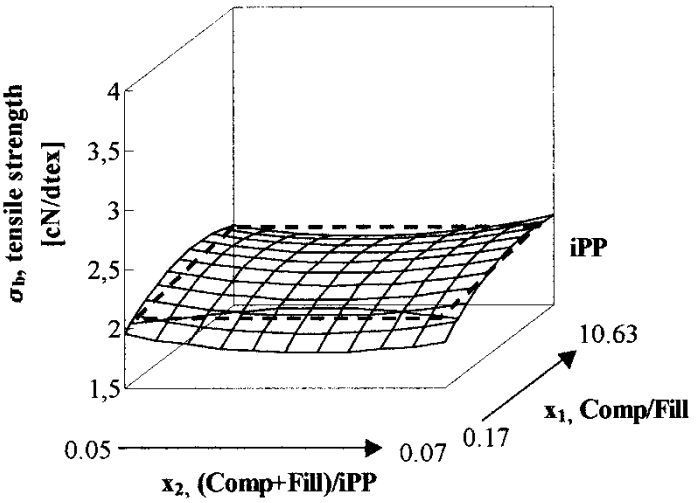

Figure 1. Influence of factors of the method "Experimental Design" x_1 (Comp/Fill) and x_2 ((Comp + Fill)/iPP) on the tensile strength of nanocomposite fibers for drawing ratio $\lambda = 3$.

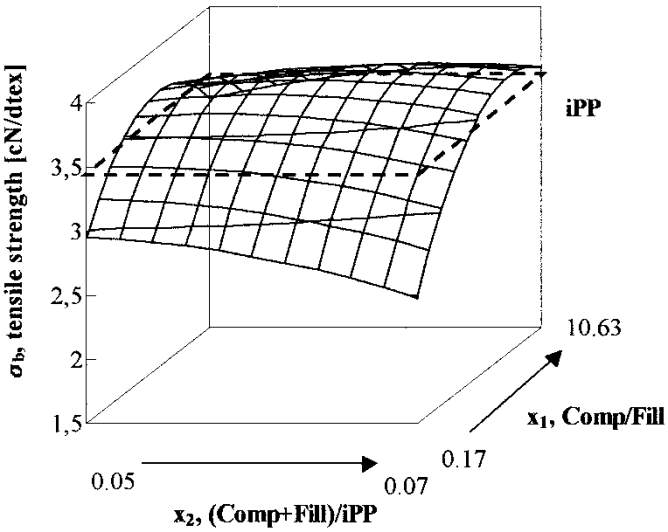

Figure 2. Influence of factors of the method "Experimental Design" x_1 (Comp/Fill) and x_2 ((Comp + Fill)/iPP) on the tensile strength of nanocomposite fibers for drawing ratio $\lambda = 4$.

strength of fibers does not change in the investigated composition range (Figure 3). From this observation, it follows that at a suitable content of filler, in the concentration interval of 0.34 to 1.5 wt%, there will be no decrease of mechanical properties of the fibers. If the iPP nanocomposite fibers of this series of samples contain more than 2.76 wt% of compatibilizer, the mechanical properties do not change remarkably, but a decrease of compatibilizer under the mentioned value 2.76 wt%, results in an observed decrease of mechanical properties.

From these results, it follows, that the optimal composition of iPP-Compatibilizer-Filler should be in the region of Comp/Fill ratio from 0.16 till 2.76. The concentration range of the filler, NANOFIL, is not unambiguous. From our results, it can be stated that the presence of NANOFIL of at least 1.5% will result in the tensile strength of iPP fibers

Table 10
Tensile strength at break $-\sigma_b$ of stabilized iPP nanocomposite fibers with various drawing ratios

Samples N°	iPP (wt%)	iPP-g-MA AR504 (%)	NANOFIL (%)	Comp/ Fill	σ_b (cN/dtex)		
					$\lambda = 2$	$\lambda = 3$	$\lambda = 4$
5	**94.34**	**0.81**	**4.85**	**0.16**	**0.93**	**1.62**	**2.43**
14	94.34	4.16	1.50	2.76	1.42	2.32	3.87
15	94.34	5.03	0.63	8.03	1.40	2.34	3.77
16	94.34	5.26	0.40	13.1	1.54	2.32	3.89
17	94.34	5.32	0.34	15.7	1.59	2.27	3.79
18	100	—	—	0	1.21	2.04	3.46

Note: Values typed in bold are used from the Table 3.

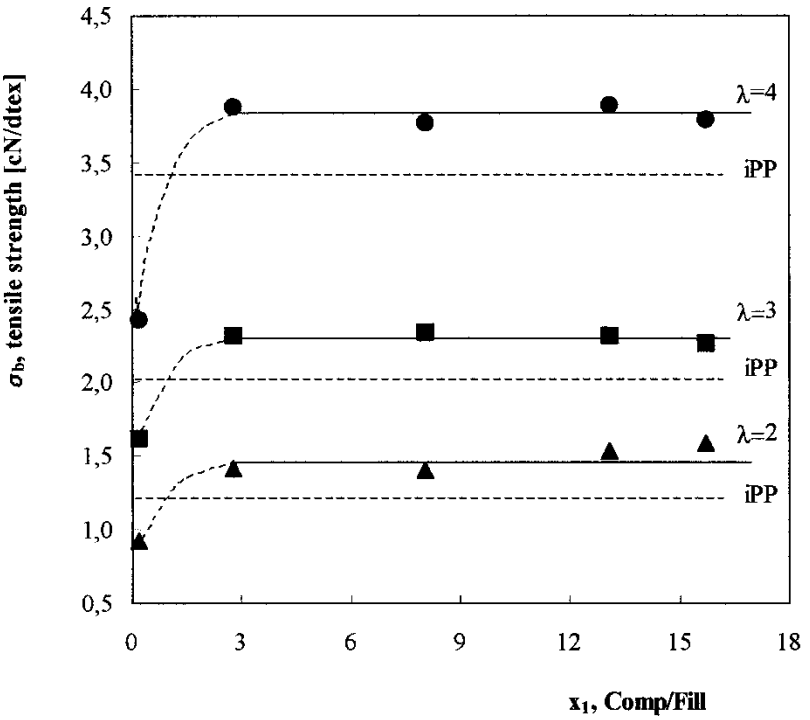

Figure 3. Dependence of tensile strength of iPP nanocomposite fibers on ratio of compatibilizer (iPP-g-MA) to filler NANOFIL (x_1, Comp/Fill) for drawing ratios $\lambda = 2$, 3 and 4.

reaching higher values compared to unfilled fibers. For the determination of the exact optimal compositions of the iPP nanocomposite mixture, a narrower interval of additives should be used in order for the method "Experimental Design", to give higher precision in regression equations.

Conclusions

From the tensile strength measurement of iPP/NANOFIL nanocomposite fibers, the following conclusions are drawn:

It was found that in a relatively wide concentration range of filler (0.16 till 2.76 wt% NANOFIL), a higher tensile strength of iPP nanocomposite fibers can be achieved than in unfilled iPP fibers.

The optimum of mechanical properties of the iPP nanocomposite fibers also requires an optimal amount of the iPP-g-MA compatibilizer.

The method of "Experimental Design" enables one to determine the region of optimal composition of iPP nanocomposite samples from the standpoint of achieving the desired mechanical properties.

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References

1. Manias, E., Touny, A., Wu, L., Strawhecker, K., Lu, B., and Chung, T.C. (2001) Polypropylene/montmorillonite nanocomposites. Review of the synthetic routes and materials properties. *Chem. Mater.*, 13: 3516.
2. Pavliková, S., Thomann, R., Reichert, P., Mülhaupt, R., Marcinčin, A., and Borsig, E. (2003) Fiber spinning from poly(propylene)-organoclay nanocomposite. *J. Appl. Polym. Sci.*, 89: 604.
3. LeBaron, P.C., Wang, Z., and Pinnavaia, J. (1999) Polymer-layered silicate nanocomposites: an overview. *Appl. Clay Sci.*, 15: 11.
4. Alexandre, M. and Dubois, P. (2000) Polymer-layered silicate nanocomposites: preparation, properties and uses of a new class of materials. *Mater. Sci. Eng.*, 28: 1.
5. Nam, P.H., Maiti, P., Okamoto, M., Kotaka, T., Hasegawa, N., and Usuki, A. (2001) A hierarchical structure and properties of intercalated polypropylene/clay nanocomposites. *Polymer*, 42: 9633.
6. Reichert, P., Hoffmann, B., Bock, T., Thomann, R., Mülhaupt, R., and Friedrich, Ch. (2001) Morphological stability of poly(propylene) nanocomposites. *Macromol. Rapid Commun.*, 22 (7): 519.
7. Saujanya, C. and Radhakrishnan, S. (2001) Structure development and crystallization behavior of PP/nanoparticulate composite. *Polymer*, 42: 6723.
8. Kawasumi, M., Hasegawa, N., Kato, M., Arimitsu, U., and Okada, A. (1997) Preparation and mechanical properties of polypropylene-clay hybrids. *Macromolecules*, 30 (20): 6333.
9. Svoboda, P., Changchun, Z., Wang, H., Lee, L.J., and Tomasko, D.L. (2002) Morphology and mechanical properties of polypropylene/organoclay nanocomposites. *J. Appl. Polym. Sci.*, 85: 1562.
10. López, D.G., Picazo, O., Merino, J.C., and Pastor, J.M. (2003) Polypropylene-clay nanocomposites: effect of compatibilizing agents on clay dispersion. *Eur. Polym. J.*, 39 (5): 945.
11. Alexy, P. and Viselka, M. Basis of design and evaluation of experiments and subprogram STATIS for MS EXCEL 5.xx-7.00, STU CHTF Bratislava (in Slovak).