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Application of Factorial Experimental Design in Predicting Mechanical Properties of Polypropylene based Composites

Vineta Srebrenkoska[1]
[1]Faculty of Technology, University Goce Delchev
Krste Misirkov No. 10-A
Stip, R.Macedonia

Dimko Dimeski[1]
[1]Faculty of Technology, University Goce Delchev
Krste Misirkov No. 10-A
Stip, R.Macedonia

Gordana Bogoeva Gaceva[2]
[2]Faculty of Technology and Metallurgy,
Ss.Cyril and Methodius University,
1000 Skopje, R. Macedonia

***Abstract*— The purpose of the study is to access the applicability of full factorial experimental design in predicting mechanical properties of polypropylene based composites. The polypropylene based composites reinforced with kenaf fibers were manufactured by melt mixing and consequently compression molded. For all manufactured composites the tensile and flexural strengths were determined. The preparation of the composites was conducted by applying 2^2 full factorial experimental design. The fiber/resin ratio was taken to be the first factor and the second – mixing temperature. For the first factor, low and high levels are chosen to be 20/80 and 40/60, respectively and for the second factor – 175°C and 195°C, respectively. Based on the experimental data, the regression equations were obtained which best describes the influence of the chosen factors on the mechanical properties of the composites. The first-order linear model to approximate the response, i.e. the flexural and tensile strength and modulus of the composites within the study domain (20/80 – 40/60) ratio x (175-195) °C was used. The influence of each individual factor to the response function is established, as well as the interaction of both factors. It was found that the estimated first-order regression equation with interaction gives a very good approximation of the experimental results of the mechanical properties of composites within the study domain.**

The polypropylene based composites exhibit good characteristics for application as construction materials for housing systems.

Keywords*— *experimental design, polypropylene, kenaf fibers, melt mixing

I. INTRODUCTION

Factorial experimental designs are used frequently in industry, especially in various stages of product development and in process and quality improvement. It has the advantage of saving time and money in running the experiment. The factorial experimental design may be run for one or more of the following reasons: to determine the principal causes of variation in a measured response, to find the conditions that give rise to a maximum or minimum response, to compare the responses achieved at different settings of controllable variables and to obtain a mathematical model in order to predict future responses. The full factorial experimental design allows to make mathematical modeling of the investigated process in a study domain in the vicinity of a chosen experimental point [1].

In recent years, the development of composites from biodegradable or recycled polymers and natural fibers have attracted great interests [2]. Amongst polymer composites, special attention has been given to polypropylene (PP) composites, due to their added advantage of recyclability [3]. As an oil-based product, PP could not be classified as a biodegradable polymer, but by introducing thermo-sensitive catalysts to increase the degradability, PP takes an important place in composite materials. Many investigations have been made on the potential of natural fibers as reinforcements for polymer composites and in several cases the results have shown that they exhibit good stiffness and promising properties [4-9]. Natural fibers (NF) reinforced materials offer target environmental advantages such as reduced dependence on non-renewable energy/material sources, lower pollution and greenhouse emission [3, 4]. Processing of these composites offer easy and cost-effective processes, so the market for these composites seems to be promising and realizable for double-digit growth in the near future [5–7]. Natural lignocellulosic fibers (flax, jute, hemp, etc.) represent an environmentally friendly alternative to conventional reinforcing fibers (glass, carbon) [8]. Depending of their performance, when they are included in the polymer matrix, lignocellulosic fibers can be classified into three categories: (1) wood flour particulates, which increase the tensile and flexural modulus of the composites, (2) fibers of higher aspect ratio that contribute to improving the composites modulus and strength when suitable additives are used to regulate the stress transfer between the matrix and the fibers, and (3) long, natural fibers with the highest efficiency amongst the lignocellulosic reinforcements. The most efficient natural fibers have been considered those that have a high cellulose content coupled with a low microfibril angle, resulting in high filament mechanical properties [10].

The purpose of the work reported in this study was to predict the mechanical properties of PP composites reinforced with kenaf fibers, compounded by reactive blending, and consequently compression molded, by applying the factorial experimental design in preparation of the samples.

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II. EXPERIMENTAL

Maleated polypropylene (PP) KA 805, produced by Montell, Italy, was used as a matrix, while the kenaf fibers, average length 5.1mm and average diameter 21µm, were kindly supplied by Kenaf Eco Fibers Italia S.p.A. (Guastall-Italy). In order to promote the matrix/fiber compatibilization, the amount of 5 wt. % of maleic anhydride grafted polypropylene (MAPP) has been added during the reactive blending. The kenaf fibers (KF) were vacuum-dried for 24h to adjust the moisture content to 1-2 wt% before they were mixed to produce composites.

The preparation of the composites has been performed by melt mixing, in a Brabender-like apparatus (Haake Rheocord, New Jersey, USA). During the blending the coupling agent MAPP has been added. First, the polymer and the coupling agent were mixed and then the fibers have been added. In 2^2 full factorial experimental design (FFED) that was used, the fiber/resin ratio is taken to be the first factor and the second factor– mixing temperature.

The fiber content in all composites was 20 wt% and 40wt%. The kneading (mixing) temperature was 175°C and 195°C, and the mixing proceeded for 10 minutes with progressively increasing the mixing speed up to 64 rpm. With that assumption, we have taken the first-order linear model with interactions to predict the response function, i.e. the mechanical properties of the composites within the study domain (20/80 – 40/60) ratio x (175-195) °C.

The obtained composites were cut into pellets to produce sheets for further characterization.

The FFED allows to make of mathematical modeling of the investigated process in a study domain in the vicinity of a chosen experimental point [1]. In order to include the whole study domain, we have chosen the central points of both ranges to be experimental points. For the fiber content of the composites we have chosen the experimental point to be 30 wt%, and for the kneading temperature the experimental point – 185 °C (which corresponds to previously defined levels). In accordance to the FFED procedure 4 (2^2) trails are needed, i.e. all possible combinations of the variables are tested. The coding of the variables is conducted in accordance with Table 1.

TABLE I. CODING CONVENTION OF VARIABLES

	Fiber content, wt%	Mixing temperature, °C
Zero level, $x_i = 0$	30	185
Interval of variation	10	10
High level, $x_i = +1$	40	195
Lower level, $x_i = -1$	20	175
Code	x_1	x_2

The samples for mechanical testing were fabricated by compression molding. The pellets obtained after melt mixing were put in a molding frame with desired dimensions and have been molded by thermo-compression, with increasing pressure up to 4500 Pa. After the expiration of the heating time, the press was cooled by circulating cold water. From all composites plates with thickness 3 mm were produced.

The obtained composites have been characterized by flexural and tensile tests. The tensile testing was performed according to ASTM D 638-99 standard on a Instron Universal Machine Model 4301 using unnotched samples while the flexural testing was conducted according to ASTM D 790 standard using three point flexural sharply notched samples. The tests were performed at crosshead speed of 2mm/min, at room temperature. Each result obtained represents the average of five samples.

III. RESULTS AND DISCUSSION

The test results (flexural and tensile properties) for the polypropylene based composites are presented in Table 2 together with the experimental matrix.

TABLE II. EXPERIMENTAL MATRIX WITH RESULTS

Trials	x_1	x_2	$x_1 x_2$	Tensile test		Flexural test	
				Stress at peak, MPa	Modulus, MPa	Stress at peak, MPa	Modulus, MPa
1	-1	-1	+1	26,7	2875	42,6	1941
2	+1	-1	-1	36,8	2881	48,5	2575
3	-1	+1	-1	33,9	3034	44,8	1884
4	+1	+1	+1	42,4	2416	51,8	2531
-1 Level	20	175					
+1 Level	40	195					

By implementing the 2^2 full factorial experimental design, it was found that response functions for flexural (y_{fk}) and tensile strength (y_{tk}) with coded variables, are

$$y_{tk} = 34.95 + 4.65x_1 + 3.2x_2 - 0.4x_1x_2 \quad (1)$$

$$y_{fk} = 46.925 + 3.225x_1 + 1.375x_2 + 0.275x_1x_2 \quad (2)$$

and in engineering or natural variables, y_{fn} *and* y_{tn}:

$$y_{tn} = -38.2 + 0.465x_1 + 0.32x_2 \quad (3)$$

$$y_{fn} = 11.8125 + 0.3225x_1 + 0.1375x_2 \quad (4)$$

In the FFED the term x_1x_2 is the interaction between factors which also might have influence on the response, in our case stress at peak values for tensile and flexural tests.

Analyzing the regression equations it can be found out that the main positive contribution to the mechanical characteristics is given by the fiber content of the composites i.e. tensile and flexural strength are directly proportional to the fiber content of the composites. On the other hand, the kneading temperature of the composites has lower positive effect on strengths. The interaction of the two factors, for the tensile and flexural tests, with coefficient of -0,4 and +0, 275 respectively to the tensile and flexural tests, has a neglegible negative and positive effect on the response which is of secondary order compared to the influence of fiber content and mixing temperature. So, the interaction of the two factors can be omitted in the regression equations with engineering variables.

The overall results for the mechanical properties of polymer composites based on polypropylene reinforced with kenaf fibers are presented in Table 2. The increased kenaf fibers content in the composites resultes in higher flexural modulus from 1941 MPa for EPP/KF (80/20) to 2575 MPa for EPP/KF (60/40). Obviously, at higher fiber content, the interfacial area between the fiber and the polymer also increases which results in increase of the interfacial bonding between the kenaf fibers and polypropylene. For regular shape reinforcements, the strength of the composites increases due to the reinforcement to support stress transfer from the polymer matrix [9,10].

To validate the implementation of the FFED in the study and the assumed model, theoretically calculated results are compared with experimental values for composites with fiber content of 20, 25, 30, 35 and 40 wt% manufactured at fixed mixing temperature of 185ºC.

This comparison can be done with any other value for the fiber content or mixing temperature as long as it is within the study domain. The results are presented in Figure 1.

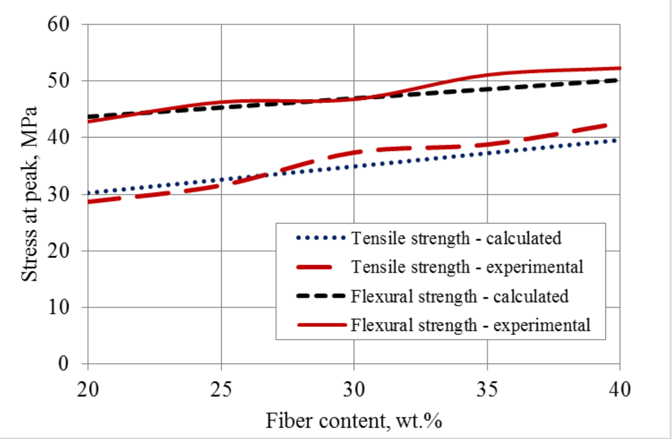

Fig. 1. Mechanical properties vs. fiber content of the composites

As it can be seen from Figure 1 there is a good agreement between calculated and experimental values. All calculated values are placed in a straight line which is in accordance to the assumed model of the experiment and are in close proximity of the experimental data.

Based on the obtained regression equations in engineering or natural variables we could do the design of composites. So, for a given request for tensile or flexural strengths, by substitution of y_{fn} *and* y_{tn}, in the equations (3) and (4), the fiber content of the composites can be calculated and then the appropriate fiber/matrix ratio will be used in fabrication of the composites. Also, y_{fn} *and* y_{tn} i.e. values of tensile or flexural strengths, can be calculated for a given fiber content (x_1 factor). In both above cases the mixing temperature has to be fixed at 185ºC for the most favorable outcome.

IV. CONCLUDING REMARKS

Experimental measurements of mechanical properties of polypropylene based composites for determined ranges of fiber/matrix ratio and mixing temperatures have been carried out implementing 2^2 full factorial experimental design. A regresion equations were established for flexural and tensile strengths as a function of the fiber/matrix ratio and the mixing temperatures of the composites. Very good agreement has been found between experimental and calculated values. It was observed that if the study domain is precisely established (narrow enough), the full factorial experimental design can be employed to give good approximation of the response, i.e. stress of peak values.

Based on the results obtained for mechanical properties, the flexural and tensile properties are higher of the composites with higher fiber content. PP based composites with kenaf fibers, represent a good potential for utilization as ecologically friendly materials for different products.

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