



Effects of extruding factors on mechanical and physical properties of polypropylene/rubberwood flour composites

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Abstract

The objective of this research was to investigate effects and interactions of extruding factors on the mechanical and physical properties of composites produced from recycled polypropylene and rubberwood flour. Central composite design (CCD) and response surface methodology (RSM) were applied to study the effects of temperatures in zones 1 and 2, as well as screw rotation speed of a twin-screw extruder. The results revealed that an increase of temperature in zone 1 resulted in increased tensile strength (TS) and hardness, but the modulus of rupture (MOR) and water absorption (WA) of the composites were reduced. Increased temperature in zone 2 decreased the TS and MOR of the composites, but the hardness and WA clearly increased. Furthermore, the TS, MOR and WA of the composites decreased with an increase of screw rotation speed, but hardness increased.

Keywords: Wood-plastic composites, Rubberwood flour, Extruder machine, Central composite design

1. Introduction

Nowadays, wood-plastic composites (WPCs) have growing applications in several industries such as construction, infrastructure, automobiles, and boat building, among others [1]. WPCs offer low maintenance, low density, low cost, resistance to water absorption and fungal activity, recyclability and eco-friendliness [2]. WPCs can be produced by adding wood sawdust and additives into molten plastic matrices [3]. The primary manufacturing processes for WPCs are injection molding, compression and extrusion [4]. However, extrusion is the more popular process for manufacturing building materials for decking, fencing, ceilings, flooring, and wall panels, among others due to its continuous linear profile.

Extrusion is the primary way of manufacturing WPCs [4]. It involves mixing of a compounded wood-polymer composite and forcing the melted materials through a die into a shape that has a continuous profile. It was then cut to desired lengths. Furthermore, in manufacturing WPCs, the extrusion conditions and factors, such as temperature and screw speed, are important variables affecting the final material properties, as well as the production capacity and quality of products.

Salleh et al. (2014) [5] reported that composites produced with high extrusion temperatures showed better tensile strength and elastic modulus, compared to low temperature extruded materials. Phonngam et al. (2013) [6] found that melt pressure and pressure at the die head were reduced with increased extruder temperature, but increased with increasing screw speed. Lewandowski et al. (2011) [7] revealed that increasing extrusion temperature in range of 140 to 185 °C improved the tensile strength of polyvinyl chloride (PVC) composites. Thongpradid et al. (2008) [8] reported that the extrudate swell ratio increased with an increasing screw rotation speed, while increasing temperature reduced shear viscosity due to lower restrictions in chain molecule movement. Despite some research in the area of effects of the extruding factors, no studies were found in the literature on interactions between temperature and screw rotation speed of a twin-screw extruder and their effects on the physical and mechanical properties of WPCs, analyzed using statistical techniques.

The effects of raw material characteristics on the mechanical, physical and thermal properties of extruded WPCs have been extensively studied. However, there are few investigations on effects of the extrusion factors in twin-screw extruders on the properties of WPCs. Furthermore, this research is needed to understand the interactions between

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temperature and screw speed during extrusion, as well as to optimize extrusion factors of polypropylene/rubberwood flour composites. The main objective of this work was to investigate the effects and interactions of the extrusion factors on the mechanical and physical properties of composites from recycled polypropylene and rubberwood flour using central composite experimental design. The results of this work will facilitate WPC manufacturing for improving production capacity and quality of these products.

2. Materials and methods

2.1 Materials

Recycled polypropylene (rPP) pellets under the trade name WT170 were purchased from Withaya Intertrade Co., Ltd (Samutprakarn, Thailand). Rubberwood flour (RWF) was obtained from a local furniture industry (Songkhla, Thailand) and used as a lignocellulosic filler. Prior to mixing, the rubberwood flour was sieved through an 80 mesh sieve and dried in an oven at 110 °C for 8 h. Maleic anhydride-grafted-polypropylene (MAPP), chosen as a coupling agent for modifying the interfacial adhesion between filler and plastic matrix, was supplied by Sigma-Aldrich. An ultraviolet (UV) stabilizer under the trade name, MEUV008, was purchased from TH Color Co., Ltd (Samutprakarn, Thailand).

2.2 Experimental design

Central composite design (CCD) was employed to model conditions with three independent variables to evaluate the effects and interactions of extrusion factors on the mechanical and physical properties of WPCs. The independent variables included the temperatures in zones 1 and 2, as well as the screw rotation speed. CCD is particularly helpful in sequential experiments because it can often create on previous factorial experiments and model a nonlinear response variable by adding axial and center points [9]. The selected variables and their levels are shown in Table 1. The levels were chosen based on the results from preliminary investigations and a literature review. The CCD experimental design, statistical analysis and response surface methodology (RSM) were done with Design-Expert software (Version 8.0.6, Stat-Ease, Inc.).

According to CCD experimental design, 20 model scenarios including 14 different conditions and 6 replicate conditions of center points were randomly arranged according to Design-Expert software, as shown in Table 2. When the response data were obtained from the testing, a regression analysis was performed to determine the coefficients of the response model [10], and then RSM was done.

Table 1 Selected factors and their variable levels for the experimental design

Factor	Coded-variables level				
	-1.68	-1	0	+1	+1.68
A; Temp. 1 (°C)	173	180	190	200	207
B; Temp. 2 (°C)	173	180	190	200	207
C; Speed (rpm)	16	50	100	150	184

Note: Temp. 1: Temperature zone 1; Temp. 2: Temperature zone 2; Speed: Screw rotation speed.

Table 2 Experimental conditions and responses based on central composite design

Run	Factor			Response			
	Temp. 1 (°C)	Temp. 2 (°C)	Speed (rpm)	TS (MPa)	MOR (MPa)	Hardness (Shore D)	WA (%)
1	190	190	100	26.05	46.24	73.50	1.19
2	200	180	50	24.75	52.98	73.50	0.90
3	180	200	150	20.61	44.38	73.50	1.77
4	190	190	16	26.40	48.64	71.90	1.49
5	190	190	100	23.69	44.75	71.70	1.39
6	180	200	50	26.57	42.13	72.50	2.07
7	190	173	100	25.19	46.01	72.20	0.47
8	180	180	150	23.94	42.93	73.50	1.40
9	190	190	100	25.67	44.87	72.90	1.98
10	207	190	100	25.76	48.76	74.30	0.97
11	173	190	100	23.22	49.99	71.40	2.23
12	200	200	150	23.94	48.22	74.80	1.26
13	190	190	100	24.93	44.25	73.40	1.34
14	200	180	150	28.44	39.70	73.80	0.59
15	190	207	100	22.26	42.14	74.10	1.53
16	190	190	100	25.76	43.70	72.90	1.25
17	190	190	184	22.34	43.17	74.30	1.14
18	180	180	50	26.63	51.86	71.80	1.71
19	200	200	50	25.57	48.64	74.20	1.10
20	190	190	100	24.03	45.30	74.10	1.33

Note: TS: Tensile strength; MOR: Modulus of rupture; WA: Water absorption.

2.3 Composite processing

The 55 wt% rPP and 45 wt% RWF were melt-blended into WPC pellets using a twin-screw extruder (Model CTE-D25L40 from Chareon Tut Co., Ltd, Samutprakarn, Thailand). The barrel temperatures of the extruder were controlled in range 130–190 °C, while the screw rotation speed was controlled at 50 rpm. The extruded WPC strands were then blown to cool them and was subsequently pelletized. After that, the 95.9 wt% WPC pellets, 3.9 wt% MAPP and 0.2 wt% UV stabilizer were dry-blended and fed into the twin-screw extruder, as shown in Figure 1. The extrusion temperature profile and screw rotation speed were varied following Table 2. WPC panels were then extruded through a rectangular 8 mm × 16 mm die and cooled in atmospheric air.

2.4 Characterizations

2.4.1 Mechanical tests

Mechanical tests were done using a Universal Testing Machine (Model NRI-TS500-50 from Narin Instruments Co., Ltd, Samutprakarn, Thailand). Tensile tests were performed according to ASTM standard D638. Type-IV tensile specimens with dimensions of 115 mm × 19 mm × 4 mm and cross-head speed with 5 mm/min were used. The modulus of rupture was measured using a three-point bending test, according to ASTM standard D790. Nominal dimensions of 4.8 mm × 13 mm × 100 mm, a cross-head speed of 2 mm/min and a span of 80 mm were used in testing. All the mechanical tests were done under ambient conditions at 25 °C with five replications.

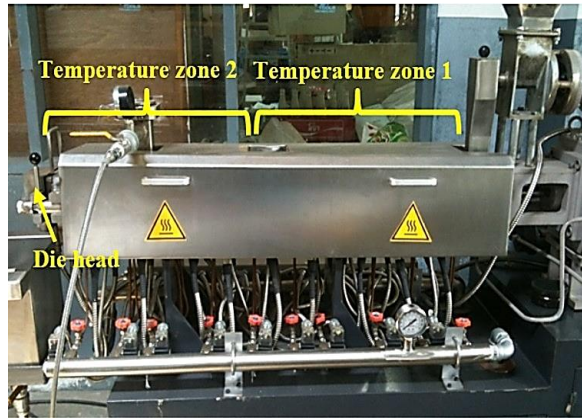


Figure 1 Barrel temperatures of the twin-screw extruder

2.4.2 Hardness test

A hardness test was performed using Durometers (Shore D scales), according to ASTM standard D2240. Specimens with nominal dimensions of 16 mm × 16 mm × 6.5 mm were used in testing. The tests were under ambient conditions at 25 °C with five replications.

2.4.3 Water absorption test

Water absorption (WA) measurements were conducted according to ASTM standard D570. Specimens with dimensions of 15 mm × 30 mm × 6 mm were used. Prior to testing, the specimens at each condition were dried in an oven at 50 °C for 24 h, and then weighed with a precision of 0.001 g and subsequently submerged in water at room temperature (25 °C). After 24 h, the immersed specimens were removed, patted dry with tissue paper and immediately weighed to determine the percentage of WA.

3. Results and discussion

3.1 Effect of extrusion factors on tensile strength

Analysis of variance (ANOVA) of response surface models revealed that the TS response was best fit with a two-factor interaction (2FI) model, rather than linear, quadratic or cubic models. This best fit model had significant sequential model sums of squares (p -value = 0.0008) and insignificant lack of fit (p -value = 0.7381), meaning that this model performs well [11]. The ANOVA results in Table 3 indicate that 2FI model is statistically significant. The effect of the extrusion factors (A: Temp. 1, B: Temp. 2 and C: speed) showed their significance on the TS (p -values less than 0.05). However, speed had the highest effect on the TS of WPCs. Furthermore, the TS was also affected by significant interactions between Temp. 1 and speed, Temp. 2 and speed, but the interaction between Temp. 1 and Temp. 2 was insignificant.

In application of the RSM, all the significant coefficients were used to create a regression equation, as shown in Table 4, which presents the relationship between TS and the other extrusion factors. This equation had a coefficient of determination (R^2) of 0.8484, adjusted coefficient of determination ($\text{adj-}R^2$) of 0.7942 and a predicted coefficient of determination ($\text{pred-}R^2$) of 0.6538. This indicates that 84.84% of the total variability in data could be explained by this models. R^2 values close to 1 indicate good fit [12]. The $\text{pred-}R^2$ value of TS was 65.38%, meaning that this fitted model was unable to explain about 34.62% of the variability in new predicting information [11].

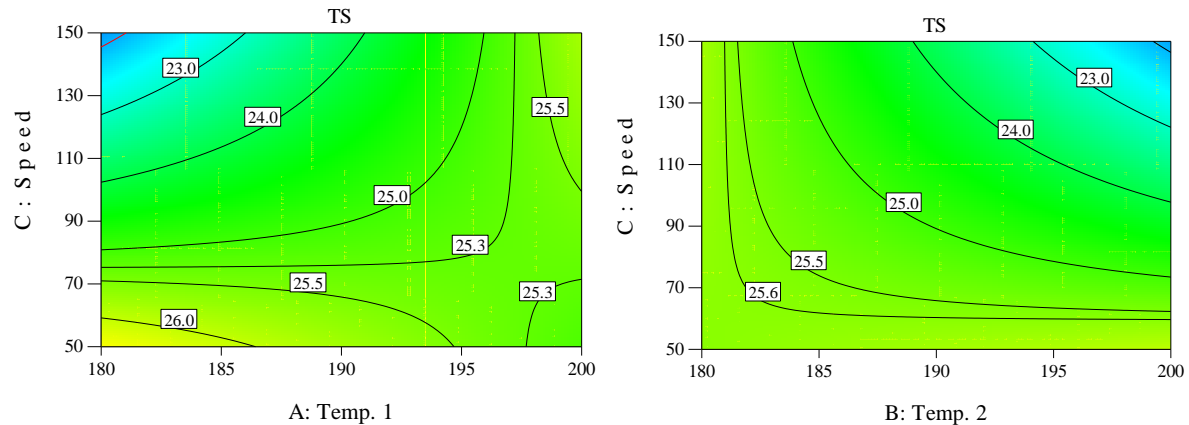
Table 3 Analysis of variance for each model

Source	TS		MOR		Hardness		WA	
	F-value	P-value	F-value	P-value	F-value	P-value	F-value	P-value
Model	12.14	<0.0001*	27.97	<0.0001*	13.39	0.0001*	59.63	<0.0001*
A	8.47	0.0121*	7.69	0.0197*	20.94	0.0003*	356.62	<0.0001*
B	14.34	0.0023*	9.52	0.0115*	6.72	0.0197*	108.46	<0.0001*
C	17.94	0.0010*	74.00	<0.0001*	12.51	0.0027*	23.81	0.0006*
AB	0.01	0.9066	22.42	0.0008*	-	-	0.44	0.5230
AC	19.51	0.0007*	7.12	0.0236*	-	-	4.73	0.0547
BC	12.55	0.0036*	83.44	<0.0001*	-	-	5.15	0.0466*
A ²	-	-	43.42	<0.0001*	-	-	23.09	0.0007*
B ²	-	-	1.11	0.3161	-	-	10.70	0.0084*
C ²	-	-	2.51	0.1442	-	-	0.09	0.7628
Lack of Fit	0.62	0.7381	3.59	0.0933	0.30	0.9552	1.13	0.4491

*P-value less than 0.05 was considered significant.

Table 4 Regression equation for each property of wood-plastic composites

Property	Regression equation	R^2	Adj- R^2	Pred- R^2
TS	$40.68448 - 0.20023A + 0.1269B - 0.12035C + 0.0026775AC - 0.0021475BC$	0.8484	0.7942	0.6538
MOR	$1272.55852 - 8.80701A - 3.63793B - 0.85177C + 0.015575AB - 0.001755AC + 0.00601BC + 0.016034A^2$	0.9465	0.9153	0.8001
Hardness	$50.57054 + 0.072324A + 0.040971B + 0.011183C$	0.7151	0.6617	0.6039
WA	$18.40001 - 0.4002A + 0.25167B - 0.024775C + 0.00012BC + 0.000952589A^2 - 0.000638401B^2$	0.9721	0.9592	0.9318

**Figure 2** Contour plots for the effects of the extrusion factors on tensile strength

The regression equation of the TS response was also employed to generate the contour plots shown in Figure 2. These plots reveal the TS values varying from 23.0 to 26.0 MPa. With Temp. 1 values greater than 195 °C, speed ranging from 90 to 150 rpm, TS was not significantly affected. At temperatures below 195 °C, the TS of WPCs slightly decreased with increasing speed. This was due to a greater shear force resulting in increased heating, degradation of the wood and decreasing molecular weight of the plastic [13]. With screw speeds over 80 rpm, increasing Temp. 1 enhanced the TS of WPCs, but the TS was slowly reduced with increasing Temp. 1 values when the screw speed was less than 80 rpm. Likewise, at high screw speeds, the TS of WPCs was clearly reduced with increasing Temp. 2 values. The temperature in zone 2 of the extruder is important for the process of extrusion. If this zone is at too high a temperature, degradation of wood and the plastic matrix occurs [14].

3.2 Effect of extrusion factors on modulus of rupture

ANOVA analysis indicated that data for MOR best fit a quadratic model. The sequential model sums of squares (p -value = 0.0004) is significant, and the lack of fit (p -value = 0.0933) was insignificant. In Table 3, the quadratic model was shown significant by the ANOVA. Likewise, the ANOVA revealed that Temp. 1, Temp. 2 and speed significantly affected the MOR of WPCs, but that the speed showed the highest effect on the MOR. The interaction terms between Temp. 1 and Temp. 2, Temp. 1 and speed, and Temp. 2 and speed significantly affected the MOR of WPCs.

In Table 4, the regression equation for MOR response of WPCs indicated high values of R^2 (0.9465), adj- R^2 (0.9153) and pred- R^2 (0.8001), which can be used in fitting parametric models [15]. The contour plots from quadratic regression models are displayed in Figure 3. They indicate that the MOR values varied in range of 42.0 to 50.0 MPa. An

increase in Temp. 1, from approximate 180 to 188 °C, resulted in a reduction of MOR values. The MOR clearly increased with increasing temperature. This is because of greater mixing efficiency at high temperatures. Zone 1 of the extruder is where mixing occurs [16]. So, at a higher mixing temperature, better dispersion of wood particles into the plastic matrix occurred, resulting in minimal voids and enhancement of mechanical properties. Homkhiew et al. (2105) [17] revealed that the mechanical properties of WPCs depend on voids in their structure, dispersion of fibers in its matrix and interfacial adhesion between wood flour and matrix plastic. WPCs with few voids, good dispersion and strong interfacial bonding achieved good mechanical properties. Furthermore, when the screw speed was less than 110 rpm, an incremental increase in Temp. 2 decreased the MOR values of WPCs. The MOR values of WPCs were steadily reduced with increased screw speed. Potential mechanisms causing these trends are explained above.

3.3 Effect of extrusion factors on hardness

The data for the hardness of WPCs were best fit using a linear model. The ANOVA analysis indicates that the linear model is statistically significant (p -value = 0.0001), as shown in Table 3. The ANOVA also revealed that the effects of Temp. 1, Temp. 2 and speed on hardness are significant. No statistically significant interaction effects on the hardness were indicated. In Table 4, the linear regression equation for the hardness of WPCs had low values of R^2 (0.7151), adj- R^2 (0.6617) and pred- R^2 (0.6039). However, the pred- R^2 of 0.6039 is in reasonable agreement with the adj- R^2 of 0.6617.

Figure 4 displays contour plots showing the effect of the extrusion factors on hardness from linear regression models. The hardness values in the contour plots were variable in range of 72.0 to 74.0. Increased Temp. 1 and Temp. 2 values in range of 180 to 200 °C linearly increased the hardness of WPCs. This is because a consistent melt of the plastic matrix

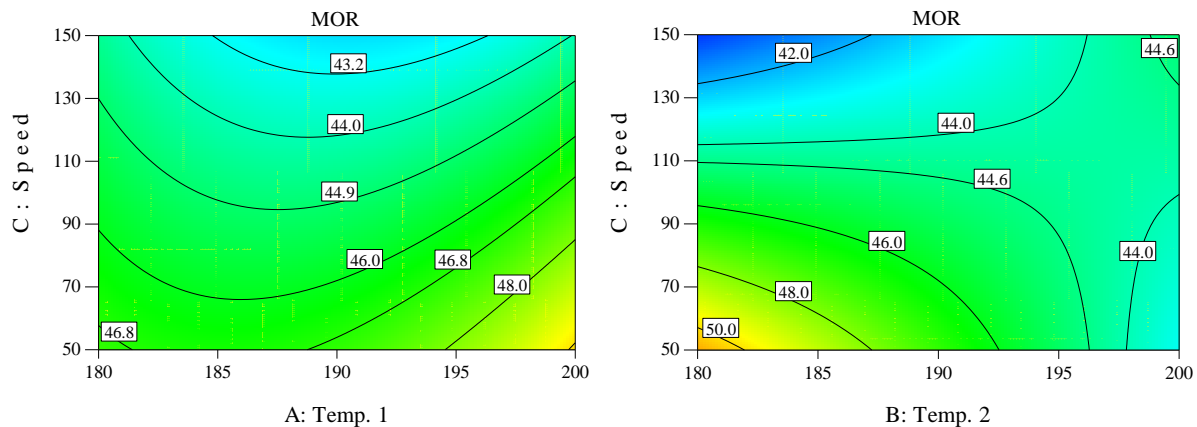


Figure 3 Contour plots for the effects of the extrusion factors on modulus of rupture

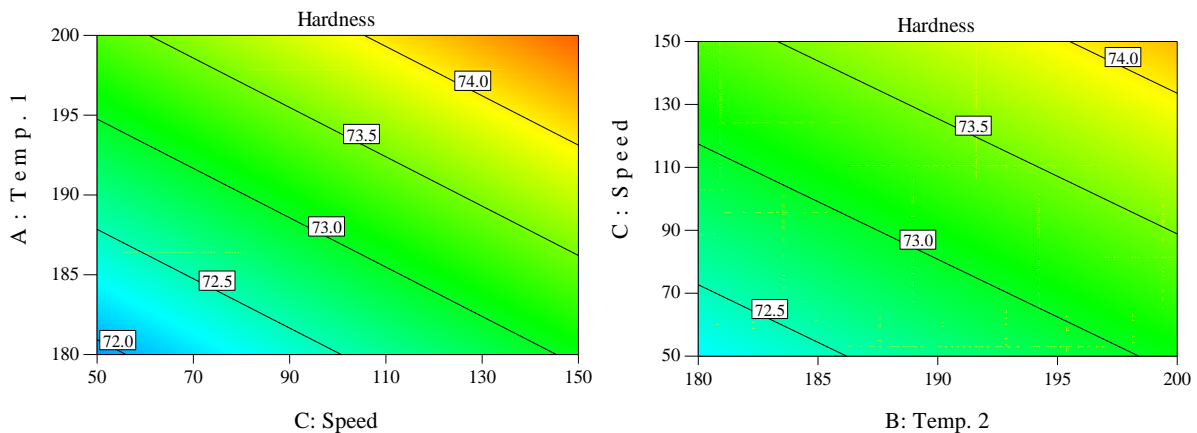


Figure 4 Contour plots for the effects of the extrusion factors on hardness

led to a uniform dispersion of the wood flour in the composites [5, 18], and eventually increased hardness. Likewise, the hardness of WPCs clearly increased with increasing screw speed. This is due to the incremental increase in extrusion force at the die head of extruder, resulting in a harder surface for the extrudate WPCs.

3.4 Effect of extrusion factors on water absorption

The WA data were best fit with a full quadratic model due to significant sequential model sums of squares (p -value = 0.0010) and insignificant lack of fit (p -value = 0.4491) as well as maximizing the adj- R^2 (0.9652) and pred- R^2 (0.9004) values. In Table 3, the quadratic model was statistically significant as shown by ANOVA analysis. All of the extrusion factors significantly affected the WA response, whereas the Temp. 1 value showed the highest effect on the WA of WPCs, considering its largest f -value (356.62). Furthermore, the WA of WPCs was also affected by a significant interaction term between Temp. 2 and speed, while the interaction terms between Temp. 1 and Temp. 2, Temp. 1 and speed were statistically insignificant.

The quadratic regression equation shown in Table 4 for the WA response of WPCs indicates high values of R^2 (0.9721), adj- R^2 (0.9592) and pred- R^2 (0.9318), further indicating good fit. It was also observed that the pred- R^2 value of the adjusted quadratic model was higher than the full quadratic model previously presented. The contour plots for WA from the quadratic regression model are displayed in Figure 5. The WA of WPCs were slightly reduced with

increased speed from 50 to 150 rpm with Temp. 1 in range of 180 to 200 °C. The reason for this is probably related to a consistent melt of plastic leading to uniform dispersion of the wood flour in the composites [5, 18]. When WPCs have uniform spatial distribution and strong interfacial bonding between the components, entry of water into the structure is more difficult [11]. Sathishkumar et al. [19] concluded that four factors accelerate of water absorption in WPCs. They are nature of the wood lumen, hydrophilicity of wood cellulose, micro-cracks in the wood flour, and adhesion between wood fibers and the plastic matrix. Increasing Temp. 2 slowly enhanced the WA of WPCs. An excessively high temperature resulted in degradation of the wood-plastic matrix [20]. Thus, voids in the WPCs structure were enhanced, which resulted in higher WA.

3.5 Optimizing the extruding condition of WPCs

An optimal extrusion condition for WPCs was determined to maximize TS, MOR and hardness while minimizing WA. Design-Expert software was employed to optimize multi-objective regression models. It generated a desirability score that balances the fitted models. The contour plot in Figure 6 displays a desirability score (0.682) under conditions considered optimal. The desirability score was quite low because the uncontrolled variability in data of TS and hardness was high. The optimal extrusion conditions were found to be Temp. 1 of 200 °C, Temp. 2 of 180 °C and an 81 rpm screw speed, as given in Table 5 with their predicted responses. Table 5 also reveals the average

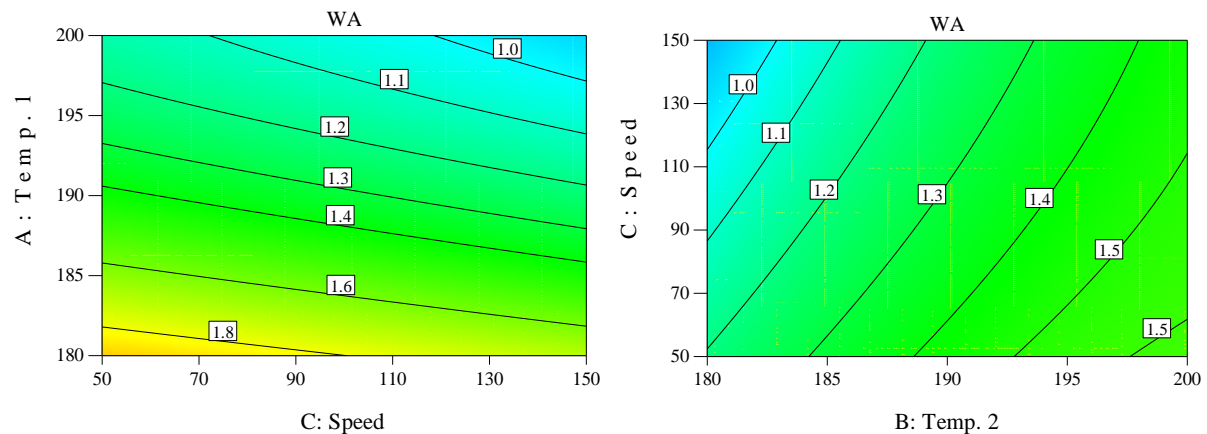


Figure 5 Contour plots for the effects of extrusion factors on water absorption

Table 5 Predicted responses from the optimal extrusion condition of WPCs

	Factor			Predicted Response				Desirability Score
	Temp. 1 (°C)	Temp. 2 (°C)	Speed (rpm)	TS (MPa)	MOR (MPa)	Hardness (Shore D)	WA (%)	
Predicted	200	180	81	25.83	48.61	73.32	0.82	0.682
Observed				24.45	49.59	74.50	0.86	
				(1.92)*	(2.24)	(0.45)	(0.14)	

* The values in parentheses are standard deviations of five replicates.

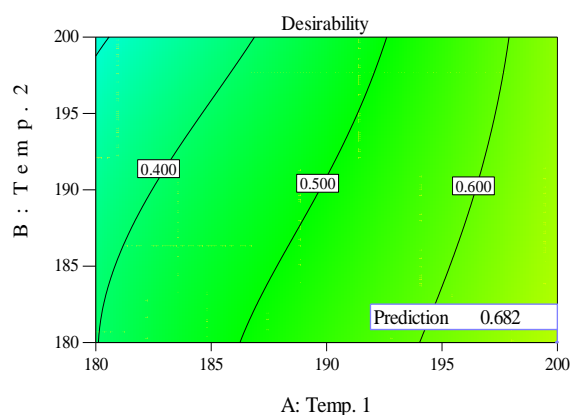


Figure 6 Desirability plot for the optimal extrusion conditions for WPCs

experimental material properties at this optimal extrusion condition. It was found that the maximum deviation between the experimental average and the model prediction occurred for TS, which was less than 6%.

4. Conclusions

The composites from recycled polypropylene and rubberwood flour were manufactured using a twin-screw extruder. Design and analysis of a CCD experiment were used to understand the effects and interactions of extrusion factors for WPCs. Extrusion factors, such as the temperatures in zones 1 and 2 and the screw rotation speed, significantly affected the mechanical and physical properties of WPCs. Production with a higher temperature in zone 1 of the extruder improved the TS, hardness and WA of WPCs. Better dispersion of wood flour into the plastic matrix occurred at a higher temperature. An increase of temperature in zone 2 reduced the TS and MOR, whereas the hardness and WA of

WPCs were clearly increased. Additionally, increasing screw rotation speed degraded the TS and MOR, but improved the hardness and WA of the WPCs. This occurred because of increased extrusion force at the die head, resulting in harder surfaces. Higher temperature in zone 1, lower temperature in zone 2 and moderate screw rotation speed are suggested for the manufacture of WPCs.

5. Acknowledgements

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