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Minimizing the creep of recycled polypropylene/rubberwood flour composites with mixture design experiments

Chatree Homkhiew¹, Thanate Ratanawilai¹ and Wiriya Thongruang²

Abstract

Composites of rubberwood flour (RWF) and recycled polypropylene (rPP) were produced into panel samples by using a twin-screw extruder. The effects on creep behavior of mixture fractions of rPP, RWF, maleic anhydride-grafted polypropylene (MAPP), and ultraviolet (UV) stabilizer were studied in a D-optimal mixture design. Creep was significantly affected by the composition. Increasing the fraction of RWF decreased creep, while MAPP and UV stabilizer increased it. The models fitted were used to optimize a desirability score that balanced multiple creep characteristics. The model-based optimal formulation 50.5 wt% rPP, 44.9 wt% RWF, 3.5 wt% MAPP, 0.1 wt% UV stabilizer, and 1.0 wt% lubricant was experimentally validated to have low creep closely matching the model predictions.

Keywords

Wood-plastic composites, recycled polypropylene, rubberwood flour, mixture experimental design, creep behavior

Introduction

Wood-plastic composites (WPCs) have been extensively developed and used in non-structural applications.¹ For example, WPCs are increasingly used to replace softwood lumber in deck building, to improve durability.^{2,3} The advantages of WPCs include high specific strength and stiffness, resistance to water absorption, and positive impact on environmental issues. These have stimulated the development of WPC materials for also structural applications.^{1,4,5} However, these composites are poorly suited for some applications due to long-term creep under loading. This study is aimed to evaluate and improve the creep characteristics of specific WPCs.

Waste materials locally available in southern Thailand were used as raw materials because of both environmental benefits and low cost. Rubberwood (*Hevea brasiliensis*) waste is mainly produced by sawmills and furniture industry, both prevalent in southern Thailand. Of their total wood intakes, these industries generally generate about 34% wood wastes and about 54% rejects of plantation wastes, while only 12% of the

rubberwood ends up in the products.⁶ Most of the wood waste can be used in medium-density fiberboard and particle board.⁷ However, the use of wood waste as reinforcement in plastic composites is of great interest, with environmental and economic benefits. The advantages of wood particles include biodegradability, low health hazard during handling, and non-abrasive nature,^{8,9} when substituted for synthetic fillers such as glass fiber, carbon fiber, and other inorganic fillers. In addition, plastic waste is one of the major constituents of global municipal solid waste.¹⁰ In 2008, at least 33.6 million tons post-consumer plastics were generated in the USA, of which 28.9 million tons went to landfills,

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2.6 million tons to combustion and energy recovery, and only 2.2 million tons to recycling^{11,12}—only a tiny fraction of plastic waste is recycled. Blending post-consumer plastics with wood flour to create value-added products could increase the value of plastic waste and impact its reuse practices.¹¹ Plastic waste is a promising raw material for WPCs because of low cost¹⁰ and properties similar to virgin materials. For example, composites made from recycled high-density polyethylene (rHDPE) have similar or, in some cases, better mechanical properties than composites made from virgin HDPE.^{13,14} The mechanical properties of composites are not better with virgin polystyrene than with recycled polystyrene.¹⁵ Ashori¹⁶ studied the potential of municipal solid waste materials for making wood plastic composites. Waste wood and paper can replace inorganic fillers in thermoplastic composites, and these composites can be reclaimed and recycled repeatedly. Ashori and Sheshmani¹⁷ made hybrid composite materials with a combination of recycled newspaper fiber (RNF) and poplar wood flour as reinforcement, and with recycled polypropylene (rPP) as the polymer matrix. They found that the composites with a high fraction of RNF had high water absorption. Madhoushi et al.¹⁸ studied the effects of sanding dust loading and nanoclay content on the physical and mechanical properties of polypropylene. Addition of sanding dust significantly decreased tensile and flexural properties of the composites, and flexural, tensile, and withdrawal strength of fasteners were improved by the addition of 2 wt% nanoclay in the matrix. Nourbakhsh and Ashori¹⁹ evaluated the effects of the fiber content and compatibilizing agent concentration on the mechanical properties and water absorption of composites from poplar fiber and rHDPE. The compatibilizer polyethylene-grafted maleic anhydride improved the flexural properties that now increased with wood content. In another study, no statistically significant differences were found in mechanical properties of composites, on comparing recycled plastics (HDPE and polypropylene) with virgin plastics.²⁰ Polypropylene waste and wood waste are promising alternative raw materials for making low cost WPCs.²¹ To reduce solid waste disposal in landfills and have low cost WPC products¹³ with good mechanical properties and low creep deformation, suitable WPC formulations need to be developed.

Design of experiments contributes to efficiently finding the best formulations. Typical designs include Taguchi method, factorial design, and mixture design.²² The fractions of components in a mixture cannot be changed independently because they must add up to 100%, and mixture designs make use of this fact.²² A D-optimal mixture experimental design allows to fit models that can be used to optimize the

formulation of a composite material.²³ It also allows placing restrictions on the formulations, such as lower or upper limits on the fractions of some components.^{23,24} Mixture designs have recently been applied in food and pharmaceutical industries to find optimal formulations because they appear efficient in providing useful models with a comparatively small number of experiments. However, prior studies on WPCs seem not to have used D-optimal mixture designs. A four-factor central composite design was applied to develop a response surface model and to study the foamability of rigid PVC/wood-flour composites.²⁵ A 2⁴ factorial design was used to determine the effects of two hindered amine light stabilizers (HALS), a colorant, an ultraviolet absorber, and their interactions, on the photostabilization of wood flour/HDPE composites.²⁶ A Box-Behnken design with response surface method was adopted to determine which variables influenced board performance significantly.²⁷ In the current study, a D-optimal mixture design was used to model the creep of WPCs. The ultimate goal of this work was to optimize the composite formulation using rPP and rubberwood flour (RWF) for minimal creep.

Materials and methods

Materials

rPP pellets, with a melt flow index of 11 g/10 min at 230°C, were purchased from Withaya Intertrade Co., Ltd (Samutprakarn, Thailand). RWF, used as a natural reinforcement, was collected from a local furniture factory (Songkhla, Thailand). Its chemical composition (by dry weight) was cellulose 39%; hemicellulose 29%; lignin 28%; and ash 4%.⁶ The interfacial bonding between wood flour filler and polymer matrix was also modified, using maleic anhydride-grafted polypropylene (MAPP) with 8–10% of maleic anhydride, supplied by Sigma-Aldrich (Missouri, USA). HALS additive under the trade name MEUV008, chosen as the ultraviolet (UV) stabilizer, was supplied by TH Color Co., Ltd (Samutprakarn, Thailand). Paraffin wax, chosen as the lubricant (Lub), was purchased from Nippon Seiro Co., Ltd (Yamaguchi, Japan).

Experimental design to optimize formulation

The D-optimal design of mixture experiments was created with Design-Expert software (version 8.0.6, Stat-Ease, Inc.) to statistically evaluate and model the effects of component fractions on creep properties and to optimize the formulation. The optimal experimental design of WPC formulations specified the component fractions of rPP (x_1), RWF (x_2), MAPP (x_3), UV (x_4), and Lub (x_5). The upper and lower limits of experimental range

Table 1. Constraints for the mixture design of experiments.

Component	Proportion restriction (wt%)
rPP (x_1)	$50 \leq x_1 \leq 70$
RWF (x_2)	$25 \leq x_2 \leq 45$
MAPP (x_3)	$3 \leq x_3 \leq 5$
UV stabilizer (x_4)	$0 \leq x_4 \leq 1$
Lub (x_5)	$= 1$

Table 2. Experimental compositions in mixture experimental design and measured responses.

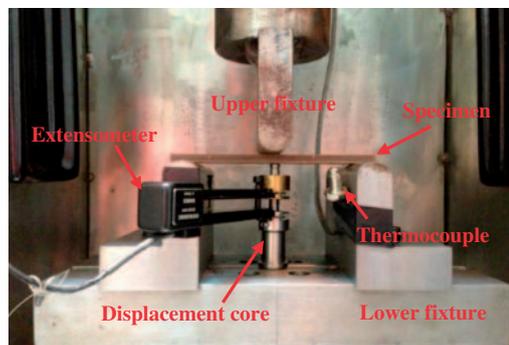
Run no.	Mixture proportion (wt%)					Creep strain (%)		
	x_1	x_2	x_3	x_4	x_5	C_e	C_{ve6000}	C_{t6000}
1	63.9	29.9	4.5	0.7	1.0	0.98	0.33	1.31
2	70.0	25.0	3.0	1.0	1.0	1.03	0.42	1.45
3	50.0	43.0	5.0	1.0	1.0	0.77	0.27	1.04
4	54.9	38.9	4.5	0.7	1.0	0.75	0.25	1.00
5	59.5	34.5	5.0	0.0	1.0	0.80	0.37	1.17
6	55.4	39.9	3.5	0.2	1.0	0.74	0.29	1.03
7	59.5	34.5	4.0	1.0	1.0	0.78	0.31	1.09
8 ^a	59.5	34.5	5.0	0.0	1.0	0.89	0.38	1.27
9	50.0	44.3	4.3	0.5	1.0	0.67	0.27	0.94
10	68.0	25.0	5.0	1.0	1.0	1.07	0.43	1.50
11	50.0	45.0	3.0	1.0	1.0	0.70	0.25	0.95
12 ^a	50.0	43.0	5.0	1.0	1.0	0.71	0.36	1.07
13	60.3	35.3	3.0	0.5	1.0	0.88	0.34	1.22
14	64.9	30.4	3.5	0.2	1.0	0.90	0.38	1.28
15 ^a	70.0	25.0	3.0	1.0	1.0	1.03	0.40	1.43
16	51.0	45.0	3.0	0.0	1.0	0.70	0.32	1.02
17 ^a	51.0	45.0	3.0	0.0	1.0	0.69	0.26	0.95
18 ^a	50.0	45.0	3.0	1.0	1.0	0.73	0.25	0.98
19	70.0	25.0	4.0	0.0	1.0	0.94	0.42	1.36
20	69.0	25.0	5.0	0.0	1.0	0.96	0.33	1.29

^aDuplicate experiment.

for the compositions are shown in Table 1. Despite the fraction of Lub being held constant, it is included as a variable because it contributes to the 100% in the mixture. The total number of runs was 20, as shown in Table 2, including 15 different formulations and five duplications to evaluate reproducibility or variances.

Composites processing

Before compounding, the RWF was sieved through an 80 mesh standard sieve (particles smaller than 180 μm pass) and dried in an oven at 110°C for 8 h to minimize

**Figure 1.** Test apparatus of three-point bending creep.

moisture content. WPCs were then produced in a two-stage process. In the first stage, WPC pellets were produced: rPP and RWF were dry-blended, and then melt-blended into wood-plastic composite pellets using a twin-screw extruder machine (Model SHJ-36 from En Mach Co., Ltd, Nonthaburi, Thailand). The extrusion barrel with 10 temperature zones was controlled at 130–170°C to avoid degradation of the components, while the screw rotating speed was maintained at 70 rpm. The extruded strand passed through a water bath and was subsequently pelletized. In the second stage, WPC panels were produced: the WPC pellets were again dried at 110°C for 8 h. WPC pellets, MAPP, UV stabilizer, and lubricant compositions indicated in Table 2 were then dry-mixed and added into the feeder of the twin-screw extruder. The temperature profile for extruding was 130–190°C, with 50 rpm screw feed. Melt pressure at the die varied between 0.10 and 0.20 MPa, depending on wood flour content. Vacuum venting at nine temperature zones was also used to purge volatile compounds. The samples were extruded through a rectangular 9 mm \times 22 mm die and cooled in atmospheric air. The specimens were machined for flexural creep testing, following the standard of American Society for Testing and Materials (ASTM).

Characterization

Three-point bending creep tests of rPP/RWF composites were carried out on an Instron Universal Testing Machine (Model 5582 from Instron Corporation, MA, USA) in Figure 1, according to ASTM D2990 standard. All the tests were performed on 13 mm \times 4.8 mm \times 100 mm (width \times thickness \times length) rectangular samples, and a test span of 80 mm. Before the creep tests, the specimens were equilibrated for 15 min, and the tests were conducted at a temperature of 25°C (ambient conditions). The total time of the testing was 100 min (6000 s) under a constant stress of 19 MPa. Five replications of each formulation were tested.

Morphological analysis

The formation of cracks and interfacial morphology between the wood flour and the polymeric matrix were analyzed with a scanning electron microscope (SEM). The fracture surface of the specimen before creep testing was fractured in liquid nitrogen. Likewise, the fracture surface of the specimen after creep testing was fractured by the creep characterization. SEM imaging was performed using a FEI Quanta 400 microscope (Oregon, USA) at an accelerating voltage of 20 kV. The samples (fracture surfaces) before and after creep tests were sputter-coated with gold to prevent electrical charging during the observation. Specimens were imaged at magnifications of 150 \times and 2500 \times .

Results and discussion

The D-optimal mixture design of experiments, with five fractions as (mutually dependent) variables (that sum to one), had 20 runs in a randomized order. The three determined responses were the values of the instantaneous creep strain (C_e), of the viscoelastic creep strain after 6000 s (C_{ve6000}), and of the total creep strain after 6000 s (C_{t6000}), and the results are summarized in Table 2.

Statistical analysis of the response surface model

The data for C_e , C_{ve6000} , and C_{t6000} were fit with linear models by multiple linear regression, with no statistical need for quadratic, special cubic, and cubic models. For example, a summary of modeling the C_{t6000} response is shown in Table 3. The sequentially fit linear model is significant (p -value less than $\alpha=0.05$), but the higher order terms are not. The adjusted coefficient of determination ($adj-R^2$) and predicted coefficient of determination ($pred-R^2$) shown in Table 4 have fairly good values at 0.8780 and 0.8413, respectively. The values in Table 4 came from an analysis of variance (ANOVA) on the significant effects relative to the creep responses. The ANOVA shows statistical

Table 3. Fit summary of C_{t6000} response.

Source	Sequential p -value	Lack of fit	$Adj-R^2$	$Pred-R^2$	
		p -value			
Linear	<0.0001*	0.1125	0.8780	0.8413	Suggested
Quadratic	0.2092	0.1415	0.9045	0.7242	
Special cubic	0.2454	0.1161	0.9279	-13.77	
Cubic	0.1161	-	0.9497	-	Aliased

* $P < 0.05$ indicates that model terms are significant.

Table 4. P -values from analysis of variance and model adequacy indicators for each modeled response.

Source	C_e (%)	C_{ve6000} (%)	C_{t6000} (%)
Model	Linear	Linear	Linear
	<0.0001*	0.0006*	<0.0001*
Linear mixture	<0.0001*	0.0006*	<0.0001*
Lack of fit	0.3331	0.3590	0.1125
R^2	0.9143	0.6555	0.8973
Adj- R^2	0.8982	0.5909	0.8780
Pred- R^2	0.8742	0.4296	0.8413
CV (%)	4.99	12.03	5.52

* $P < 0.05$ indicates that model terms are significant.

significance of these linear models, indicated by p -values less than α ($\alpha=0.05$). This result implies that each modeled output, C_e , C_{ve6000} , and C_{t6000} , was significantly affected by at least one of the four controlled variables. The R^2 value for C_{ve6000} is relatively poor, partly because its determination was “noisy” with a high CV.

The R^2 values of the C_e , C_{ve6000} , and C_{t6000} are 0.9143, 0.6555, and 0.8973, indicating that 8.57%, 34.45%, and 10.27%, respectively, of the total variability in observations is not explained by the models; R^2 values close to 1 indicate good fits.²⁸ R^2 values will always increase when a variable is added to the model,²⁹ and the computed $adj-R^2$ should be close to R^2 value of the model selected. This indeed is the case for the fitted models, indicating it is unlikely that the models have insignificant terms included.³⁰ The $pred-R^2$ value of C_e was 0.8742, meaning that the fitted model is estimated to explain about 87% of variability in new cases, and this is in reasonable agreement with the $adj-R^2$ of 0.8982. For C_{ve6000} all of R^2 , $adj-R^2$, and $pred-R^2$ have relatively low or poor values, because C_{ve6000} was calculated as $C_{t6000}-C_e$ and this increased its relative inaccuracy. The coefficients of variation (CV), of C_e , C_{ve6000} , and C_{t6000} were estimated at 4.99%, 12.03%, and 5.52%, respectively, based on the residual variation. Low CV values indicate good precision of the determinations.

Model adequacy checking

Model adequacy checking is always necessary with a fitted model.³⁰ Figure 2(a) displays normal probability plots of the residuals for elastic creep strain C_e , and the visually good fit with a straight lines suggests the residuals are about normally distributed. The interpretation is that the residuals are Gaussian measurement noise, while the explanatory variables (fractions of rPP, RWF, MAPP, and UV stabilizer) explain the

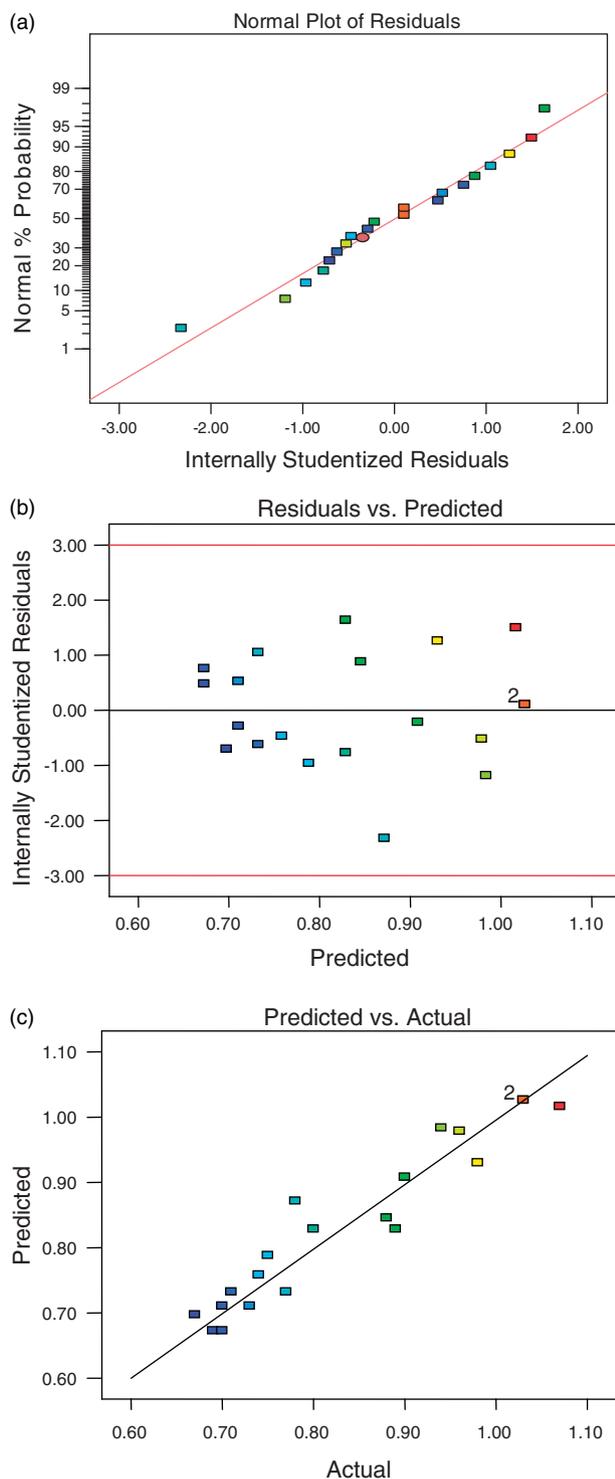


Figure 2. Model adequacy checking for elastic creep strain: (a) normal probability plot of residuals, (b) plot of residuals versus predicted values, and (c) plot of predicted versus actual values.

deterministic part of the relationship. Likewise, the presence of outliers is not strong indication, as such a failed experiment would give a large residual disabling the good straight line fit in a probability plot.²² A plot of the residuals vs. the predicted values for the model of

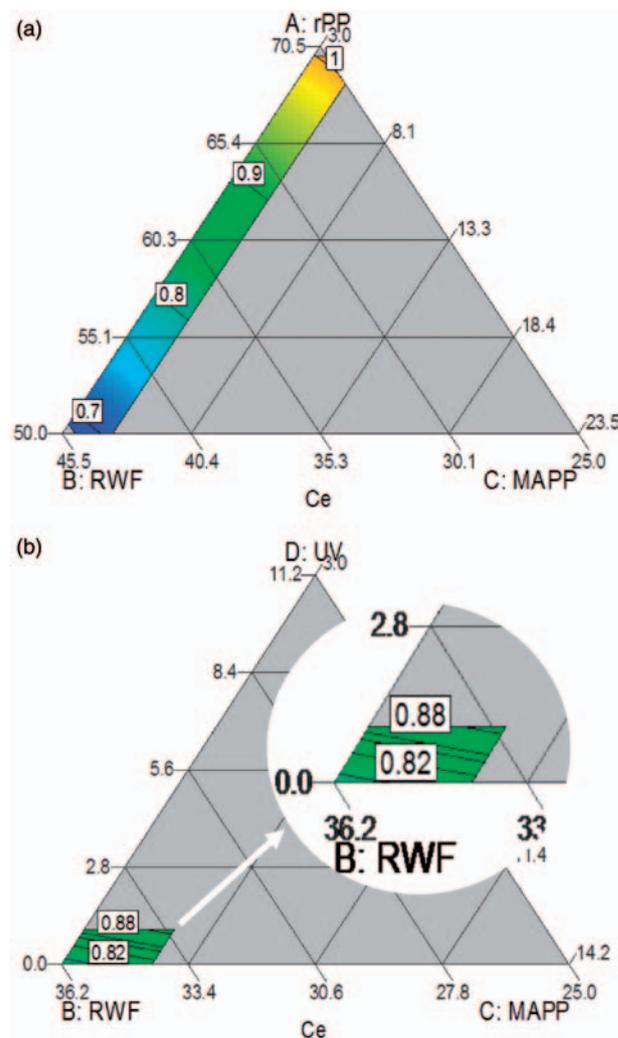


Figure 3. Triangular contour plots for effects of the compositions on elastic creep strain: (a) fixed UV stabilizer at 0.5 wt% and Lub at 1 wt% and (b) fixed rPP at 59.8 wt% and Lub at 1 wt%.

C_e is shown in Figure 2(b). There is no obvious pattern remaining, and therefore no suggestion for adding some nonlinear terms to the fit.²² Figure 2(c) shows the C_e model predictions vs. observations. The model outputs fit the actual observations quite well, with C_e model deviating from actual by less than about 5%, in alignment with the estimated CV. The model adequacy was similarly checked for C_{ve6000} and C_{l6000} , with essentially similar conclusions.

Effect of composition on the elastic creep strain, and optimal formulation

The linear regression model fitted to experimental C_e value was

$$C_e = 0.99x_1 + 0.66x_2 + 0.89x_3 + 1.78x_4 \quad (1)$$

Table 5. The optimal formulations that minimize each creep characteristic, with predicted responses.

Property (%)	Mixture proportion (wt%)					Predicted response	Desirability
	rPP	RWF	MAPP	UV	Lub		
C_e	50.0	45.0	3.9	0.1	1.0	0.67	0.996
C_{ve6000}	50.0	45.0	3.0	1.0	1.0	0.26	0.906
C_{t6000}	50.1	45.0	3.5	0.3	1.0	0.95	0.962

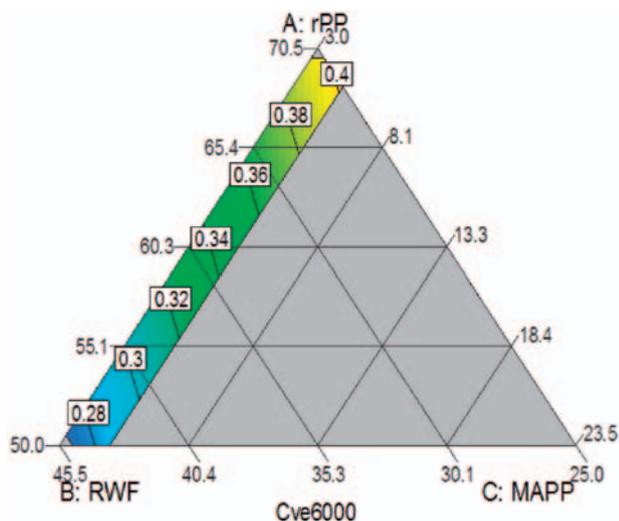


Figure 4. Triangular contour plot for effects of the compositions on viscoelastic creep strain. Constant fractions of UV stabilizer at 0.5 wt% and Lub at 1 wt%.

in which all coefficients are positive. RWF (x_2) has the smallest coefficient so it should be maximized to minimize creep. The UV stabilizer (x_4) has the largest coefficient, so its addition should be as small as possible. The experimentally covered formulations are shown in Figure 3(a) and (b), with color coding for the modeled C_e . In the triangular contour plot of Figure 3(a), the three pure components (rPP, RWF, and MAPP) are represented by the corners, while the additive levels were fixed (UV stabilizer at 0.5 wt% and Lub at 1 wt%). The contours in the colored areas, that include the experimental observations, present the C_e regression fits varying from 0.7% to 1%. The creep C_e clearly decreases with increasing RWF content. High wood flour content increases the modulus of elasticity (MOE) of composites,³¹ so that higher stress is required for the same deformation.^{7,32} The choice of MAPP content between 3 and 5 wt% barely affected the C_e . Generally, the addition of coupling agent in the WPCs decreases the creep strain due to the improved filler dispersion and improved interfacial adhesion between wood flour and polymer matrix,^{33–35} whereas

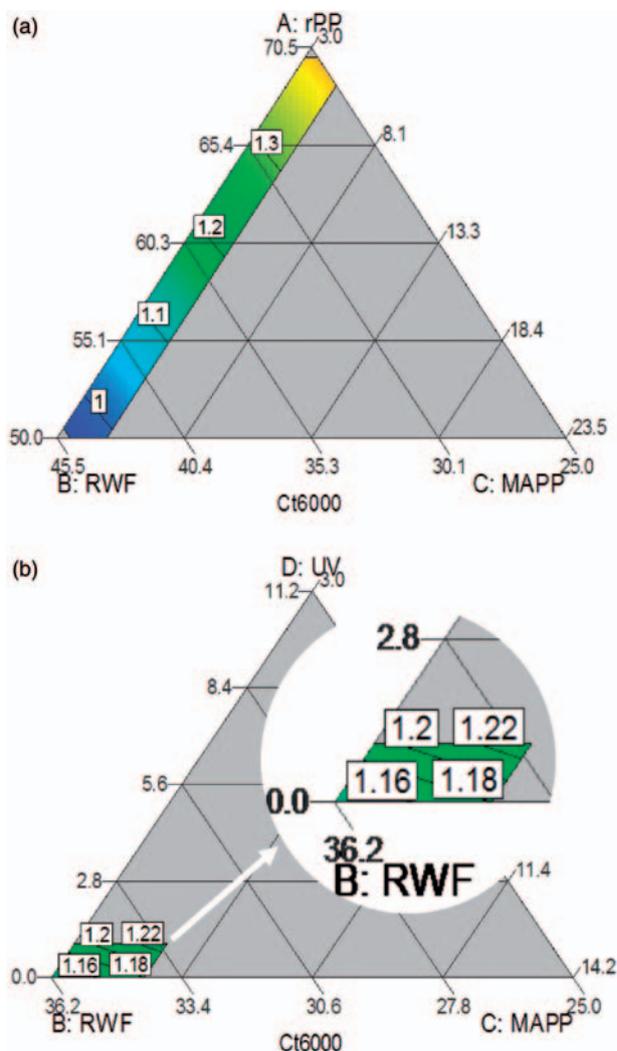


Figure 5. Triangular contour plots for effects of the compositions on total creep strain: (a) fixed UV stabilizer at 0.5 wt% and Lub at 1 wt% and (b) fixed rPP at 59.8 wt% and Lub at 1 wt%.

too much MAPP relative to wood flour will cause self-entanglement, resulting in slippage with the PP molecules.^{7,36} This slippage in the WPC structures leads to easier creep deformation of the WPC specimens. The triangular contour plot in Figure 3(b) also shows that addition of 1 wt% UV stabilizer slightly increased the elastic creep strain from 0.82% to 0.88% because UV stabilizer has some chemical reactions with the other components.³⁷ Likewise, it is known to reduce the flexural properties but to increase creep strain, due to non-homogeneous spatial distribution of wood flour, polymer, and UV stabilizer.³⁸ Therefore, the load-bearing capacity of WPC specimens decreased, resulting in increase of the creep deformation. Using 1 wt% of UV stabilizer may be unnecessary, and its fraction should be minimized to minimize creep.

The numerically optimized compositions for each creep characteristic, based on fitted models, are

shown in Table 5. In all three cases, the formulations have about 50.0 wt% of rPP and 45.0 wt% of RWF, with minor variation in MAPP and UV stabilizer fractions. Since the requirements of optimizing the different creep characteristics are not in much of a conflict, they can be approximately optimized simultaneously.

Effect of composition on the viscoelastic creep strain, and optimal formulation

The linear regression model for the viscoelastic creep strain (C_{ve6000}) was

$$C_{ve6000} = 0.40x_1 + 0.27x_2 + 0.45x_3 + 0.26x_4 \quad (2)$$

with positive coefficients. In Figure 4 C_{ve6000} (in the range of 0.28 to 0.40%) increases for high fractions

of rPP, because the mobility of polymer chains increased in the WPCs and contributed to viscoelasticity. The concentration effect of MAPP on C_{ve6000} was insignificant, similar to elastic creep strain. The optimal composition minimizing viscoelastic creep strain coincided with formulation 11, see Table 5.

Effect of composition on the total creep strain, and optimal formulation

The linear regression fit for the total creep strain (C_{t6000}) was

$$C_{t6000} = 1.39x_1 + 0.93x_2 + 1.33x_3 + 2.04x_4 \quad (3)$$

with positive coefficients. RWF (x_2) has the lowest coefficient, while UV stabilizer (x_4) had the largest

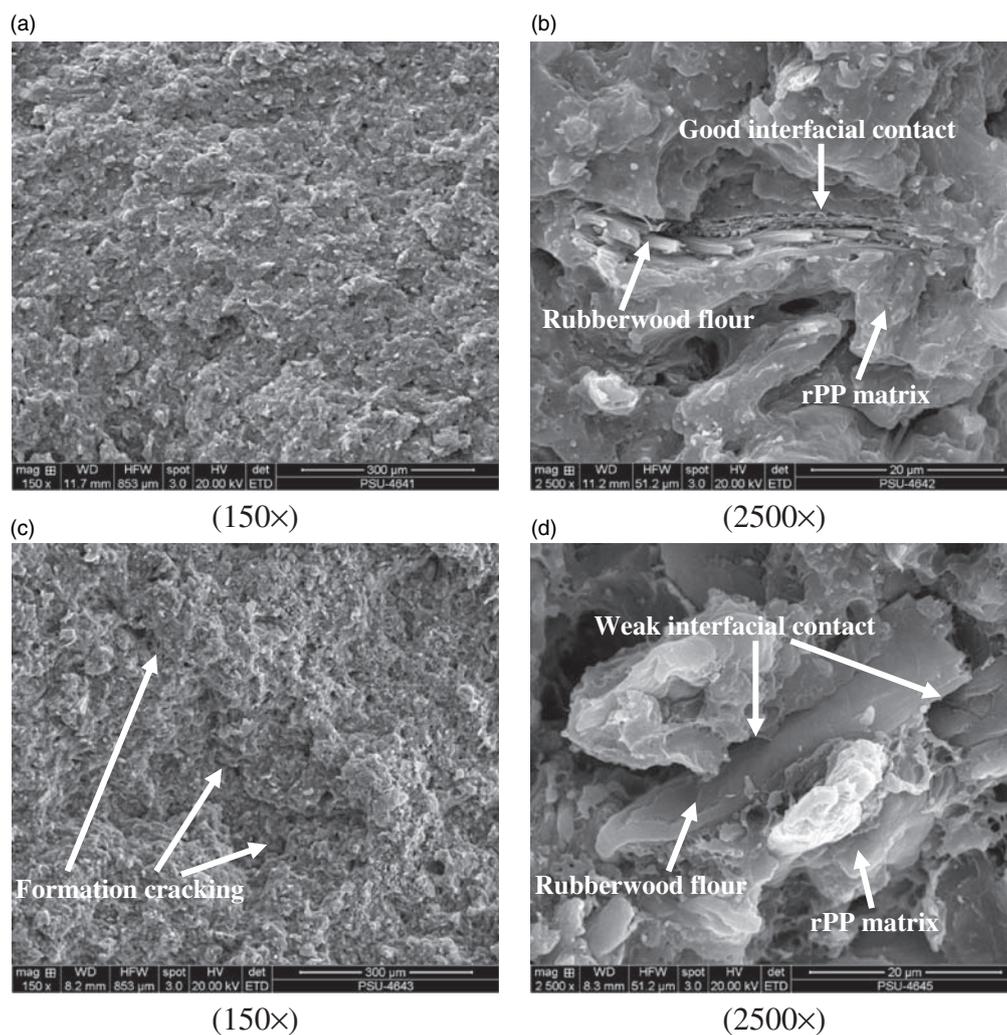


Figure 6. SEM micrographs of rPP-rubberwood flour composites showing formation of cracks and interfacial contact between wood flour and plastic matrix (Magnification 150 \times and 2500 \times): (a), (b) before creep testing and (c), (d) after creep testing.

component fractions experimentally varied, namely of rPP, RWF, MAPP, and UV stabilizer, which significantly affected all the creep characteristics (C_e , C_{ve6000} , and C_{t6000}). In general, a high fraction of RWF reduced all of these, and the optima found had 45 wt% RWF which was the maximum in the experimental design. At this wood flour loading, the modulus of elasticity was maximized, so that a comparatively high stress is required for a given creep deformation. Increasing the fraction of MAPP from 3 to 5 wt% only slightly affected the creep strain, lacking statistical significance. The addition of 1 wt% UV stabilizer slightly increased creep. The approximately optimal formulation minimizing jointly all creep characteristics was 50.5 wt% rPP, 44.9 wt% RWF, 3.5 wt% MAPP, 0.1 wt% UV stabilizer, and 1.0 wt% Lub. The joint optimization maximized a desirability score that balanced the multiple objectives, and the jointly optimal formulation was experimentally validated to produce low creep nearly as predicted.

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Conflict of Interest

None declared.

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