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Flat pressed *Pongamia pinnata* wood-flour/polypropylene composite loaded with talc: a statistical optimization

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Abstract This study was aimed to produce wood-plastic composite (WPC) from Pongamia pinnata, a biofuel producing tree species by using statistical response surface methodology (RSM). The study evaluated three parameters: (1) the mass ratio of wood and polypropylene (PP), (2) the pressing time; and (3) the talc percentage as filler, where pressure and temperature was remain constant. Maintaining wood content at 50 % or less, provided the best mechanical properties, and wood content above approximately 60 % resulted in reduction of all mechanical properties of WPCs. The results showed that pressing time has a great impact on board quality. The optimal concentration of the filler content (talc) in WPCs was 2 %. Adding the proper amount of talc can improve the mechanical properties but over-dosing affects all the properties of the WPCs.

Keywords Mechanical properties · Talc · Wood plastic composite · Statistical optimization · *Pongamia pinnata*

Introduction

Wood plastic composites (WPC) are becoming very popular over the last decade due to its structural properties and advantages relative to other competing materials (Bengtsson and Kristiina 2006; Nourbakhsh and Ashori 2009). Moreover, WPC is not susceptible to bio-deterioration under exterior and interior applications due to the possible

Md Azharul Islam iazharul@gmail.com blockage of porous sites of hydrophilic wood by hydrophobic plastic (Wechsler and Hiziroglu 2007). A lot of study demonstrated that wood-plastic ratio combination has a great effect on WPC board (Bouafif et al. 2009; Arab and Islam 2015; Al-Mamun et al. 2015). Ayrilmis and other coauthors (2011) explained that the pressing time has a vital impact on mechanical and physical properties. According to Benthien and Thoemen (2012), temperature has little effect on mechanical properties of WPC board. Different study shows that 4 mpa pressures are better for producing good quality WPC board (Min and Zhi 2004). On the other hand, melting temperature of polypropylene is ranges 130-160 °C. Therefore, in this study temperature was fixed at 180 °C and press pressure was maintained at 4 mpa. Now a day, talc is extensively used in plastic and rubber industries as fillers to increase the rigidity, creep strength and impact. Likewise, in presence of talc, wood in WPC reduces water absorption, shrinkage, deformation and creep (Martikka et al. 2012). It has been demonstrated that talc is triclinic in structure which increases the bonding capacity of polypropylene (Ross et al. 1968; Rayner and Brown 1973).

Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for developing, improving and optimizing processes and can be used to evaluate the relative significance of several affecting factors even in the presence of complex interactions (Montgomery and Douglas 2005). The main objective of RSM is to determine the optimum operational conditions for the system or to determine a region that satisfies the operating specifications (Salim et al. 2012). The techniques involved in RSM and its application on wood modification and composite materials has been well described in different informative articles (Zhao et al. 2013; Homkhiew et al. 2014; Islam et al. 2014).

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Pongamia pinnata (L.) Pierre, commonly known as Karanj, a medium-sized, evergreen, perennial and deciduous tree indigenous to the Indian subcontinent and south-east Asia, and has been successfully introduced to humid tropical regions of the world as well as parts of Australia, New Zealand, China and the USA (Scott et al. 2008). Historically, this is a multipurpose tree species used as a source of traditional medicines, animal fodder, green manure, fish poison, controlling soil erosion and fuel in India and Bangladesh (Scott et al. 2008; Allen and Allen 1981). Recently, Pongamia pinnata has been accepted and recognized as valuable source of biodiesel (Naik et al. 2008). The wood of Pongamia pinnata is white to yellowish-grey; well grained and medium to coarse textured (Allen and Allen 1981). The timber is relatively easy to saw, turn and finish, the wood is not considered a quality timber because it is not durable, tends to split and warp during seasoning and is susceptible to insect attack (Orwa et al. 2009). The wood may be used in agricultural implements, tools and combs (Das and Alam 2001).

At present, *Pongamia pinnata* is planted commercially in different areas of the world for biofuel production which also ensure abundant supply of the wood sustainably. Different wood species have different anatomical structures which governs the structural properties of WPC (Bouafif et al. 2009). Therefore, an attempt has been made in this study to prepare and testing the suitability of valuable wood plastic composite from *Pongamia pinnata* wood flour with polypropylene. This study will also determine the optimum manufacturing conditions with their corresponding responses modulus of rupture (MOR) and modulus of elasticity (MOE) to manufacture WPC using response surface methodology (RSM) as a statistical experimental design.

Materials and methods

Sample preparation for WPC production

A defect free bole of *Pongamia pinnata* tree is collected locally as raw material for the production of wood flour as lignocellulosic filler. The particles were produced using mechanical refining process without any chemical in the laboratory. The size of the wood flour ranges <700 μ m. The PP granules were also processed by a rotary grinder to make powder for evenly distribution among all the surfaces of wood particles. Before blending, wood particles with PP plastics powder were dried in a laboratory oven at 102 ± 3 °C for 24 h to reduce moisture content at 1–3 %. Talc was collected from local market.

WPC panel manufacture

After processing the raw materials, WPC composite boards was manufactured using standardized flat pressed procedures according the Box-Behnken design matrix presented in Table 1. At first, the weighted mixture was blended into a rotary drum blender. Then mats were formed manually on stainless steel caul plate of targeted dimensions of the board (6×6 inches). Teflon sheet used to avoid direct contact of plastic powder with the plate during hot pressing. Another such plate was laid up on top of the mat before inserting into the hot press (model-XLB, Manufacture no. 120,049, Qingdaoyadong, Chaina) at a constant pressure of 4 mpa. The average mat thickness of each board maintained to eight times of the targeted board thickness (6 mm). After hot pressing, the board was removed from the hot press to facilitate the setting of thermoplastic matrix for 2 days at room temperature for further processing.

Determination of composite properties

After manufacturing WPC board according to the proposed design matrix, the mechanical properties MOR (Modulus of rupture) and MOE (Modulus of elasticity) were measured by Universal Testing Machine (Model- UTN100, SR no. 11/98-2441, Fuel Instruments and Engineers Pvt. Ltd. India) according to the procedures defined in the BS (British standard) BS 1610-1:1992 (BS 1992) and IS (Indian standard) IS 1828(Part 1):1991 (IS 1991).

Experimental design

Box–Behnken design (BBD) is an independent, second order polynomial design with no treatment combinations that are extreme (Montgomery DC, Douglas C, 2005). Experiments were established based on a BBD with three factors at three levels and each independent variable were coded at three levels between -1, 0 and +1 (Table 1).

Design-Expert[®] 7.1.3 (Trial version) statistical software was used for experimental design and a second order polynomial model was used to fit the response to the independent variables is shown below-

$$\mathbf{Y} = \boldsymbol{\beta}_0 + \sum \boldsymbol{\beta}_{ii} \mathbf{X}_i + \sum \boldsymbol{\beta}_{ii} \mathbf{X}_i^2 + \sum \boldsymbol{\beta}_{ij} \mathbf{X}_i \mathbf{X}_j$$

where Y is the response (MOE or MOR), β_0 is the intercept and β_i , β_{ii} , β_{ij} are the coefficients of parameters for linear, squared and interaction effects respectively. The desirability function approach was used for the concurrent determination of optimal settings of independent variables that can determine optimum performance levels for one or more dependant variables (Islam et al. 2012).

 Table 1
 Factors, ranges and

 labels for factor Box–Behnken
 design

Factors	Name		Range and label				
			Low (-1)	Middle (0)	High (+)		
A	Ratio = Wood flou	r:PP (wt%)	1 = 70:30	2 = 60:40	3 = 50:50		
В	Talc (wt%)		0	5	10		
С	Pressing time (minute)		8	10	12		
Run	Wood flour:PP	Talc	Pressing time	MOE (N/m ²)	MOR (N/m ²)		
1 (C)	2.00	5.00	10.00	15.19	882.08		
2	3.00	10.00	10.00	15.56	614.80		
3	1.00	10.00	10.00	9.84	458.16		
4	3.00	5.00	8.00	26.88	1069.89		
5	3.00	5.00	12.00	19.9	1020.85		
6	2.00	0.00	8.00	14.17	787.19		
7	1.00	0.00	10.00	11.19	546.49		
8	2.00	0.00	12.00	15.35	630.34		
9	2.00	10.00	8.00	10.59	551.49		
10 (C)	2.00	5.00	10.00	15.43	898.76		
11	3.00	0.00	10.00	26.72	1210.01		
12	2.00	10.00	12.00	14.53	518.40		
13 (C)	2.00	5.00	10.00	16.29	975.34		
14	1.00	5.00	8.00	10.03	921.09		
15	1.00	5.00	12.00	15.64	592.81		

(C) indicates center points run for error calculation

Results and discussion

Adequacy of the Box-Behnken model

Physical and mechanical properties of the board are significantly affected by the processing parameters. Main manufacturing process parameters such as wood plastic ratio, talc percentage and pressing time were considered in this study. According to the experimental design with three processing variables each having three levels of variable, RSM Box–Behnken design yielded a total of 15 runs in a random order (Table 1). From Tables 2 and 3, it has been clearly observed from the sequential model fitting for the MOE and MOR in WPC production, the quadratic model was the best suited model in both cases. So it is needed to be determining the adequacy of the model which is evaluated through Analysis of Variance (ANOVA), normal probability plot and observed versus predicted plot for MOR and MOE.

ANOVA for MOE and MOR has been showing in Tables 4 and 5 respectively. The lack of fit (LOF) test was also conducted in ANOVA. Generally, Lack of fit (LOF) test allows us to determine if the current model adequately accounts for the relationship between the response variable and the predictors or not. If the model does not fit the data well, this will be significant. Three central points are used for calculating the pure error. In this study the value of LOF is not significant relative to the pure error regarding to MOR and MOE, which indicates a good response to the model.

From the ANOVA table co-efficient of determination (R^2) value is 0.96 for MOR and also 0.96 for MOE is highly agreement with the experimental results, indicating 96 % of the variability can be revealed by the model and are left with 4 % both of residual variability for MOE and MOR. For further soundness of the model, adjusted R^2 is used for confirming the model adequacy. The adjusted R^2 was calculated to be 0.883 and 0.896 for MOE and MOR, which indicates a good model for using in the field conditions. "Adeq Precision" measures the signal to noise ratio. It compares the range of the predicted values at the design points to the average prediction error. A ratio greater than 4 is desirable and indicate adequate model discrimination. The ratio of "Adeq Precision" for MOE and MOR are 10.67 and 12.289 respectively, indicates an adequate signal. This model can be used to navigate the design space. The model is again evaluated by observed versus predicted plot. The graphical presentation shows that the points all predicted and actual responses (Fig. 1a and b) are pointed along with 45° straight line which signifies another good reply of the model.

Source	Sequential model sum of squares									
	Sum of squares	df	Mean square	F-value	P value Prob > F	Remarks				
Mean versus total	9.091E + 006	1	9.091E + 006							
Linear versus mean	4.171E + 005	3	1.390E + 005	4.30	0.0309					
2FI versus linear	87,554.71	3	29,184.90	0.87	0.4956					
Quadratic versus 2FI	2.376E + 005	<u>3</u>	79,187.94	12.84	0.0087	Suggested				
Cubic versus quadratic	25,893.09	3	8631.03	3.49	0.2307	Aliased				
Residual	4946.72	2	2473.36							
Total	9.864E + 006	15	6.576E + 005							
Lack of fit										
Linear	3.510E + 005	9	39,001.29	15.77	0.0610					
2FI	2.635E + 005	6	43,909.49	17.75	0.0543					
Quadratic	25,893.09	3	8631.03	3.49	0.2307	Suggested				
Cubic	0.000	0				Aliased				
Pure error	4946.72	2	2473.36							
Model summary statistics	8									
Source	Std. dev.	R-squared	Adjusted R-squared	Predicted R-squared	Press	Remarks				
Linear	179.89	0.5395	0.4140	0.0961	6.988E + 005					
2FI	183.17	0.6528	0.3924	-0.5036	1.162E + 006					
Quadratic	78.54	0.9601	0.8883	0.4497	4.254E + 005	Suggested				
Cubic	49.73	0.9936	0.9552			Aliased				

Table 2 Sequential model fitting for the MOE in WPC production

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some scatter even with normal data (Fig. 2a and b). This degree of non-normality is probably not serious enough to warrant a radical change in the model.

From the statistical results, it may arrive at a judgment by reasoning that the Box–Behnken design is sufficient to predict the board strength (MOR and MOE) within the range of variables studied. The final predicted mathematical model in terms of actual significant factors for wood plastic composite board production control by different parameters are given below–

MOR $(N/mm^2) = -17.10 + 14.20 * Ratio$ +0.72 * Talc + 2.27 * Pressing time -0.49 * Ratio * Talc-1.57 * Ratio * Pressing time +0.07 * Talc * Pressing time + 2.32 * Ratio² -0.09 * Talc² + 0.04 * Pressing time² MOE $(N/mm^2) = + 254.37 - 183.59 * Ratio$ + 92.10 * Talc + 136.96 * Pressing time -25.34 * Ratio * Talc + 34.91 * Ratio * Pressing time +3.09 * Talc * Pressing time + 33.97 * Ratio² -9.81 * Talc²-12.88 * Pressing time²

Effect of processing factors on MOE

In this study, it has been observed from ANOVA for MOE (Table 4) that the linear effect of wood plastic ratio and talc, the quadratic effect of talc has significant impact on the MOE. It has been also showed that the linear interaction effect between wood plastic ratio and talc (i.e. AB) has the most significant impact on MOE. From the Fig. 3, it has been attributed that the synergistic effect of highest ratio (i.e. 50:50 wood plastic ratio) and lowest talc content produced WPC board with high MOE. It has also been observed that the MOE of compressed wood attained the highest value 1200 N/mm² for low talc content ranges 1-2.5 (on weight basis) and wood plastic ratio ranges 60:40 to 50:50. One study by the Ayrilmis and Jarusombuti (2011) stated that the MOE of WPC increased with the increase in the wood fiber content and the 50:50 ratio of wood and plastic will give better results in the application of WPC. According to Stark and Rowlands (2003), one of the primary reasons to add filler is to develop good bonding between wood-plastic and also improve the stiffness and strength of the board. In this regard we have seen within this study that using of talc as filler increases MOE. In a

Table 3 Sequential modelfitting for the MOR in WPCproduction

Source	Sequential model sum of squares										
	Sum of squares	df	Mean square	F-value	<i>P</i> value Prob > F	Remarks					
Mean versus total	3754.40	1	3754.40								
Linear versus mean			87.27	8.03	0.0041						
2FI versus linear	65.59	3	21.86	3.24	0.0815						
Quadratic versus <u>2FI</u>	<u>39.79</u>	<u>3</u>	13.26	4.68	0.0648	Suggested					
Cubic versus quadratic	13.49	3	4.50	13.44	0.0700	Aliased					
Residual	0.67	2	0.33								
Total	4135.73	15	275.72								
Lack of fit											
Linear	118.87	9	13.21	39.48	0.0249						
2FI	53.27	6	8.88	26.54	0.0367						
Quadratic	<u>13.49</u> 3 <u>4.50</u> <u>13.44</u>		0.0700	Suggested							
Cubic	0.000	0				Aliased					
Pure error	0.67	2	0.33								
Model summary st	atistics										
Source	Std. dev.	R- squared	Adjusted R-squared	Predicted R-squared	Press	Remarks					
Linear	3.30	0.6865	0.6010	0.3307	255.23						
2FI	2.60	0.8585	0.7524	0.2551	284.05						
Quadratic	1.68	0.9629	0.8960	0.4301	217.33	Suggested					
Cubic	0.58	0.9982	0.9877			Aliased					

Table 4ANOVA for responsesurface quadratic model forMOE

Source	Sum of squares	df	Mean square	F-value	Prob > F	Remarks
Model	742,200.0	9	82,467.69	13.37	0.0054	Significant
A-wood plastic ratio	243956.4	1	243956.4	98.6336	0.0099	Significant
B-talc	132917.8	1	132917.8	53.7398	0.0181	Significant
C-pressing time	40224.4	1	40224.4	16.2630	0.0563	
AB	64232.5	1	64232.5	25.9697	0.0364	Significant
AC	19493.7	1	19493.7	7.8814	0.1069	
BC	3829.1	1	3829.1	1.5481	0.3394	
A^2	4261.4	1	4261.4	1.7229	0.3197	
B ²	222232.6	1	222232.6	89.8505	0.0109	Significant
C ²	9807.9	1	9807.9	3.9654	0.1846	
Residual	30839.80	5	6167.96			
Lack of fit	25893.3	3	8631.1	3.4896	0.2306	Not significant
Pure error	4946.7	2	2473.4			
Total SS		14				

 $R^2 = 0.9601$; Adjusted $R^2 = 0.8883$; Adeq Precision = 10.676

Table 5 ANOVA for response surface quadratic model for MOR

Source

Model

B-talc

AB

AC

BC

 A^2

 B^2

 C^2

Residual

Lack of fit

Pure error

C-pressing time

0.62

8.50

13.99

0.67

7.03

5.92

0.031

13.44

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0.4664

0.0332

0.0134

0.4495

0.0454

0.0591

0.8672

0.0700

Significant

Significant

Significant

Not significant

26.88

Total SS 381.33 14

 $R^2 = 0.9628$; Adjusted $R^2 = 0.8960$; Adeq Precision = 12.289

1.76

24.06

39.63

1.90

19.90

16.76

14.16

13.49

0.67

0.088

1

1

1

1

1

1

1

5

3

2

1.76

24.06

39.63

1.90

19.90

16.76

0.088

2.83

4.50

0.33

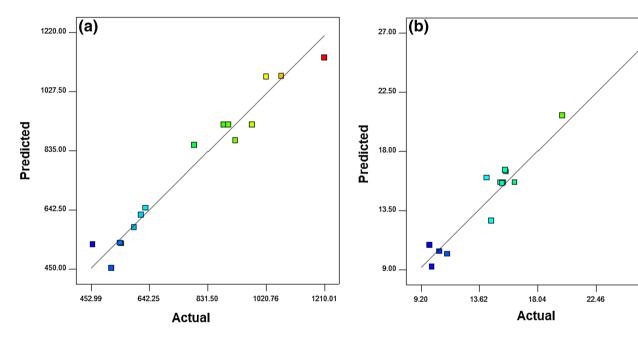


Fig. 1 Actual versus Predicted plot for a MOE b MOR

report, high quantity of talc increases thermal effect which may degrade the inner lignocellulosic component as well as MOE (Ayrilmis et al. 2011a, b).

Effect of processing factors on MOR

It has been observed from ANOVA (Table 5) for MOR that the linear effect of wood plastic ratio and talc, the quadratic effect of wood plastic ratio has significant impact on MOR. It has been also observed that the linear interaction effect between ratio and talc (AB) and wood plastic ratio and pressing time (i.e. AC) has the most significant impact on MOR. From the Fig. 4a that the MOR of wood plastic board arrived at the highest value 26.88 N/mm² for low pressing time ranges 9-10 min and high ratio of 50:50. As the pressing time start to increase from 10 to 12 min the MOR start to decrease sharply. Ayrilmis et al. (2011a, b) described in their investigation that MOR decreases with the increase of treatment time for wood plastic composite board. This strength (MOR) loss in wood is actually related to the progressive degradation of hemi-cellulose components due to increasing pressing time after a certain period of time. Loss in MOR of wood-based panels (Stark and Rowlands 2003) treated at high temperatures was reported

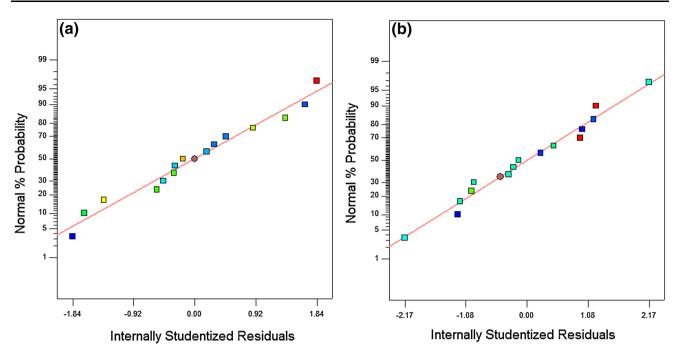


Fig. 2 Normal probability plot for a MOE and b MOR in the WPC sample population

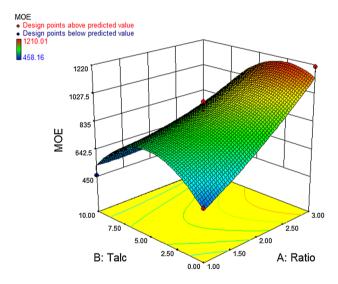


Fig. 3 3d graph of MOE as influenced by fire retardant talc and wood plastic ratio

by different authors. Similar results were also found the WPC panels in the present study.

On the other hand, as the percentage of plastic in ratio increases MOR of the boards are also increases. The highest strength of WPCs was measured when the lowest wood content is used and the strength decreased most severely when wood content increases (Leu et al. 2011; Jahanilomer and Farrokhpayam 2014). From the predicted equation of MOR, a negative interaction effect exists between pressing time and ratio. The Fig. 4a also demonstrated that the MOR starts to decrease with the increase of pressing time. It's again indicated that the combination of high pressing time and high ratio has detrimental effect on MOR.

It has been observed from the Fig. 4b that the MOR of WPC board attained the highest value 27 N/mm² for low talc content ranges 0–2.5 on weight basis and fifty–fifty wood and plastic ratio. It has been also seen that when the talc percentage is increasing from 3 to 10, it affect the MOR decreases. Talc improved chemical bond between the hydrophobic PP (polypropylene) polymer chains and hydrophilic cellulosic fibers (Kauppinen et al. 1997). Talc has significantly higher thermal conductivity (compared to the polymer), heat introduced and generated during processing is transmitted through the mixture more quickly. It could be an additional effect of the degradation of hemicellulose components for decreasing MOR. Similar observations were reported for other lignocellulosic fibers based PP (polypropylene) composites (Georgopoulos et al. 2005).

Optimization

The numerical optimization of the software has been chosen in order to find the specific point that maximizes the desirability function. The desired goal was selected by adjusting the weight or importance that might alter the characteristics of a goal. The goal fields for response have five options: none, maximum, minimum, target and within range. The criteria for the optimization of all studied factors in correspondence with MOE and MOR are shown in Table 6.

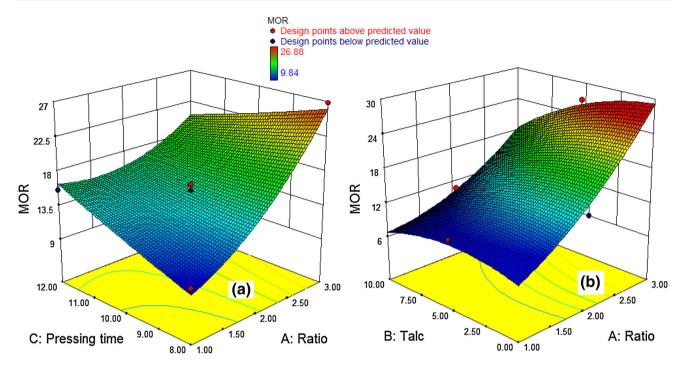


Fig. 4 3d graph of MOR a as influenced by fire retardant talc and wood plastic ratio and b by pressing time and wood plastic ratio

Table 6 Optimization of the
individual responses (di) in
order to obtain the overall
desirability response(D)

Fig. 5 Bar graph representing

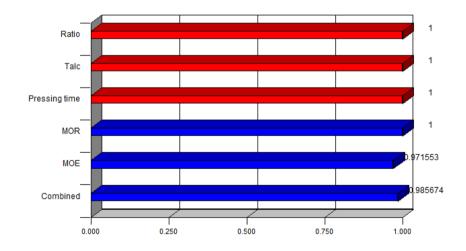
correspondence with combined

individual desirability of all

responses (di) in

desirability (D)

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
Ratio	In range	1	3	1	1	3
Talc	In range	0	10	1	1	3
Pressing time	In range	8	12	1	1	3
MOE	Maximize	458.16	1210.01	1	1	5
MOR	Maximize	9.84	26.88	1	1	5



The goal for wood plastic ratio, talc and pressing time were assigned as 'within range' with corresponding 'importance' 1. As higher MOE and MOR are usually preferred for WPC production, therefore 'maximize' for goal was selected with importance '5'. The lower limit and upper limit values of all responses are taken from the Box–Behnken design levels (Table 1). The optimization procedure was conducted under these settings and

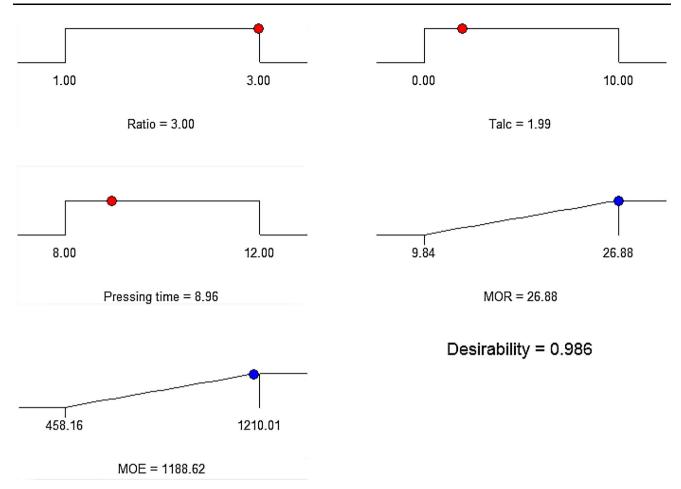


Fig. 6 Desirability ramps for numerical optimization of four selected goals

boundaries. The individual desirability functions (*di*) for each of the responses, and the calculated geometric mean as maximum over all desirability (D = 0.986) is represented in Fig. 5.

The prime objective was to maximize the MOE and MOR with recalculating all responsible factors by using desirability functions. By using this desirability function with all pre-selected goal for each factors, gave the specific value for all responses that are presented in Fig. 6. The software optimized 1188.62 N/mm² for MOE and 26.88 N/ mm² for MOR with calculating the optimized model factors of wood plastic ratio 50:50, talc 1.99 and pressing time 8.96 min for WPC production. After this optimization process of each factor levels with their corresponding responses a duplicate confirmatory experiment was conducted in order to validate the system. From the confirmation study the MOR and MOE value is obtained 28.89 and 1223.92 N/mm² respectively. This is closely related with the data obtained from desirability optimization by using Box-Behnken design.

Conclusion

In this work, particles from Pongamia pinnata along with polypropylene were used to make experimental WPC panels. Talc was used as filler to the WPC panels. Box-Behnken response surface design was successfully employed to optimize and study the individual and interaction effect of process variables such as wood-plastic ratio, pressing time and talc percentage on the WPC board. The results indicated that the process variables had a significant effect on the mechanical properties of WPC board. Model summary statistics showed that, developed model is adequate and precise with the experimental data. Analysis of variance showed a high coefficient of determination value which ensuring a satisfactory fit for the developed second order polynomial regression model with the experimental data. The optimum conditions were found to be, woodplastic ratio of 50:50, talc percentage of 1.99 % and pressing time of 8.96 min and also the predicted maximum MOR of 26.88 N/mm² and MOE of 1188.62 N/mm².

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