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EFFECTS OF AN ADDITION OF COIR-PITH PARTICLES ON THE MECHANICAL PROPERTIES AND EROSIVE-WEAR BEHAVIOR OF A WOOD-DUST-PARTICLE-REINFORCED PHENOL FORMALDEHYDE COMPOSITE

VPLIVI DODATKA KOKOSOVIH VLAKEN FENOL-FORMALDEHIDNEMU KOMPOZITU, OJAČANEM Z LESNIM PRAHOM, NA NJEGOVE MEHANSKE LASTNOSTI IN EROZIJSKO OBRABO

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Several attempts were made to investigate the effects of various process parameters on the mechanical properties and wear behavior of synthetic and natural cellulosic fibers and also particle-reinforced polymer composites. However, very few studies were carried out on the effects of various process parameters on the mechanical and wear behavior of phenol formaldehyde (PF) composites reinforced with natural cellulosic fibers and particles. Therefore, in the present study, an attempt was made to observe the effects of various process parameters on the mechanical and wear behavior of wood-dust (WD) and coir-pith (CP) particle-reinforced resole-type PF composites. First, the mechanical properties of a WD/PF composite were studied based on the content of CP particles. Then, the erosive-wear behavior of the WD/PF composite was studied with respect to five different parameters based on the Taguchi experimental design (L_{27}). The results show that the mechanical properties of the WD/PF composite was that of the composite modulus was higher than that of the composite increase with an addition of CP particles. The increment in the composite modulus was higher than that of the composite strength. The erosive test results indicate that the erosion-wear rate is affected by the particle content, impingement angle, erodent size and impact velocity. Brittle-erosion behavior was identified on the surface of the composite with a heavy erosive wear occurring at a 60° impingement angle.

Keywords: biowaste particles, phenol formaldehyde, composites, mechanical properties, erosive-wear resistance, Taguchi method

Izvedenih je bilo že kar nekaj poizkusov v zvezi z učinki različnih procesnih parametrov na mehanske lastnosti in obrabo polimernih kompozitov ojačanih s sintetičnimi in naravnimi celuloznimi vlakni in/ali delci. Toda zelo malo raziskav je bilo izvedenih glede vpliva različnih procesnih parametrov na mehanske lastnosti in obrabo fenolformaldehidnih (angl. PF) kompozitov, ojačanih z naravnimi celuloznimi vlakni in delci. Tako je v pričujočem delu predstavljen vpliv različnih procesnih parametrov na mehanske lastnosti in obrabo PF kompozitov, ki so bili ojačani z delci lesnega prahu (angl. WD) in delci kokosa (angl. CP). Te vrste kompozitov se uporabljajo za izdelavo podplatov čevljev. Najprej so bile določene mehanske lastnosti WD/PF kompozitov glede na vsebnost CP delcev. Sledili so preizkusi in analize erozijske obrabe WD/PF kompozitov glede na vsebnost (količino) delcev v kompozitu, velikost, hitrost in razdaljo učinkovanja erozijskega sredsta ter njegov vpadni kot. Preizkusi so temeljili na analizi s Taguchijevo metodo (L_{27}) s petimi različnimi parametri. Rezultati so pokazali, da se mehanske lastnosti kompozita. Erozijski testi kažejo, da je hitrost erozijske obrabe posledica vseh procesnih parametrov, to je: vsebnost delcev, udarnega kota, hitrosti in velikosti delcev izbranega erozijskega sredstva. Največja obraba zaradi erozije je bila dosežena (ugotovljena) pri 60 stopinjskem vpadnem kotu abrazijskega sredstva z nastalimi poškodbami krhkega značaja.

Ključne besede: delci bioodpadkov, fenolformaldehid, kompoziti, mehanske lastnosti, odpornost proti erozijski obrabi, Taguchi metoda

1 INTRODUCTION

Recently, polymer composites reinforced with synthetic materials have been replaced with polymer composites reinforced with bio-based natural materials. The bio-based natural materials have many advantages over the synthetic materials like renewability, biodegradability, abundant availability, low costs, etc.^{1–3} In India, particularly in the South Indian region, the biowaste materials like wood dust, coir pith, groundnut shell, coconut shell, cashew nut shell, etc. are abundantly available because in that region, coconut, groundnut and cashew nut are cultivated in large amounts. A number of timber and oil mills are also available in the Southern region of Tamilnadu, India. Therefore, bioparticles are thrown away after producing useful materials and A. SUJIN JOSE: EFFECTS OF AN ADDITION OF COIR-PITH PARTICLES ON THE MECHANICAL AND ...

dumped on the land of the village nearest to these industries.

Many studies have already reported on the properties of different bio-based natural-fiber-reinforced polymer composites in different conditions.³⁻⁷ But, only few reports are available on the properties of polymer composites filled with biowaste particles.8-11 In this investigation, an attempt was made to study the mechanical and wear behavior of PF composites reinforced with biowaste particles (WD and CP). Mechanical properties of wood-dust-particle-reinforced PF composites were evaluated based on the content of coir pith. The erosivewear behavior of the composites was studied using five different parameters such as particle content, erodent size, impingement angle, impact velocity and standoff distance. The erosive experiments were conducted as per the Taguchi experimental design. The parameters used for erosive-wear tests were also analyzed using an analysis of variance with the wear rate.

2 MATERIALS AND METHODOLOGY

2.1 Materials

Wood-dust particles were collected from the Kumar Timber and Sawmill, Karaikudi, Tamilnadu, India. Coir-pith particles were collected from the Coir Industry, Sozhavanthan, Tamilnadu, India. From the collected wood-dust and CP particles, microparticles with the average size of 800 microns were separated using a sieving machine available in our composite laboratory. The resole-type PF liquid resin was procured, together with a cross-linking agent (divinylbenzene) and acidic catalyst (hydrochloric acid), from POOJA Chemicals, Madurai, Tamilnadu, India.

2.2 Preparation of the composites

A hardboard mold box with dimensions of 150 mm × 150 mm × 3 mm was used to prepare the wood-dust and coir-pith-particle composite plates using the hand lay-up technique. Wood dust/coir pith/phenol formaldehyde composites were fabricated at three different concentrations of wood-dust and coir-pith particles, i.e., (20, 30 and 40) % mass fractions. The amount of WD particles was maintained at a fixed level of 20 % mass fraction. Three different amounts of CP particles (0–20 % mass fractions) were hybridized with the constant amount of WD particles, i.e., 20WD/0CP, 20WD/10CP, 20WD/20CP. The weight percentage of WD and CP particles and

designation of the composites are given in **Table 1**. Prior to the process, the particles were dried in sunlight for 12 h. The PF resin with the particles was mixed with a mechanical stirrer at room temperature for 30 min. Then, the cross-linking agent and acidic catalyst were also mixed into the mixture of phenol formaldehyde/particles and once again stirred with the mechanical stirrer for 15 min. After that, the mixture was poured into the mold box and allowed to cure at room temperature for 48 h.

2.3 Testing composite specimens

Composite specimens were characterized using mechanical tests such as tensile, flexural and impact tests. The tensile tests were conducted on an FIE universal testing machine (UTE 40 HGFL) in accordance with ASTM D638-10.¹² The flexural tests were performed on the same testing machine in accordance with ASTM D790-10.¹³ The impact tests were carried out on an Izod impact machine according to ISO 180.¹⁴ All the tests were conducted at room temperature and atmospheric pressure.

2.4 Taguchi experimental design

The erosive behavior of the WD/CP/PF composite was studied based on the Taguchi method and analysis of variance techniques. Experiments were performed as per Taguchi experimental design (an orthogonal array) because it is a systematic and efficient approach to get the optimum range of process parameters with a good performance. The number of experiments can be reduced due to the constructed orthogonal array, which provides a set of well-balanced experiments.¹⁵ The results obtained with this experimental design are transformed into signal-to-noise (S/N) ratios, which serve as objective functions for the optimization of parameters and help with the result analysis. There are three S/N ratios available for the optimization of several static problems: the smaller-the-better (used to minimize the response), the nominal-the-better (used whenever an ideal quality is equated with a particular nominal value.) and the largerthe-better ratio (used to maximize the response). Among these three characteristics, the minimum erosion rate comes under the smaller-the-better characteristic, which can be expressed as Equation (1):

$$S/N = -10 \text{ Log}_{10}$$
 (1)

(the mean of the sum of squares of the measured data)

Table 1: The weight percentage of WD and CP particles and designation of the composites

Total weight percentage of particles in the composites	Weight percentage of resin	Weight percentage of WD particles	Weight percentage of CP particles	Designation of composites
20	80	20	0	20WD/0CP
30	70	20	10	20WD/10CP
40	60	20	20	20WD/20CP

The five different process parameters at three levels are used in this study to observe the erosive behavior of the WD/CP/PF composite. Therefore, the actual number of experiments, based on the traditional experimental design, should be 243 (3^5). But, this number is reduced to 27 experiments using the Taguchi technique. The process parameters and their setting levels for the erosion test of the WD/CP/PF composite are presented in **Table 2**. In these experiments, the following parameters are fixed throughout the process: the type of erodent is silica, the erodent feed rate is 10.0 ± 1.0 g/min, the nozzle length is 80 mm, and the nozzle diameter is 3 mm.

Table 2: The erosive process parameters with their designation and setting levels

Process Parameters and their designation	Level I	Level II	Level III
Particle content: (A) wt%	20	30	40
Impact velocity: (B) m/sec	41	52	63
Impingement angle: (C) degree	30	60	90
Erodent size: (D) µm	300	500	700
Stand-off distance: (E) mm	80	120	160

2.5 Erosion test

The erosive tests of the WD/CP/PF composite specimens were conducted as shown in the schematic diagram of the erosion process (**Figure 1**). The main components of the erosion-test apparatus are the erodent feeder box, erodent feeder nozzle, mixing chamber, nozzle of the mixing chamber, air-flow vent, sample holder and erodent collector. Dry silica sand with three different sizes (300, 500 and 700) μ m was used as the erodent in the erosion tests. After the test, the composite samples were taken from the apparatus and cleaned with acetone.



Figure 1: Schematic diagram of the erosive process of the WD/CP/PF composite

Then, the cleaned composite specimens were dried and weighed using a precision digital balance at an accuracy of ± 0.1 mg. The composite samples were weighed before and after the erosion tests and their difference is termed as the weight loss. Then, the weight loss was recorded and used for the erosion-rate calculation. Generally, the erosion rate can be obtained as the ratio of the weight loss of samples to the weight of the eroding particle. The process was repeated until the steady-state erosion was reached.

3 RESULTS AND DISCUSSION

3.1 Mechanical properties of the composites

Mechanical tests were carried out on the WD/CP/PF composites and their results are presented in Figure 2a. The neat-resin sample had a tensile strength of 29.8 MPa, tensile modulus of 1168.4 MPa, flexural strength of 34.7 MPa, flexural modulus of 1257.4 MPa, and impact strength of 1.24 KJ/m². It can be seen that the tensile strength and modulus of the PF composite increase with an increase in the particle content. The tensile strength of the 20WD/PF composite is almost the same as that of the neat-resin sample. It shows that the addition of WD particles enhances the strength of the PF composite. The WD/PF composite without the addition of CP particles has a tensile strength of 30.4 MPa and this value increases to 41.7 MPa with the incorporation of 10 % mass fraction of CP particles; after that, it decreases to 36.8 MPa with the addition of 20 % mass fraction of CP particles. This may be due to a poor interfacial bonding between the particles and the matrix, i.e., a weak transfer of stress. Moreover, the stress concentration in the PF matrix may be created due to the corner edges of the irregularly shaped WD and CP particles.

Due to the addition of 10 % mass fraction and 20 % mass fraction of CP particles, the tensile strength of the WD/PF composite increases by about 37.17 % and 21.1 %, respectively. **Figure 2a** also shows the tensile-modulus values of the WD/CP/PF composites with respect to the particle content. The composite also reached the tensile-modulus value of the neat-resin sample with the particle addition of 20 % mass fraction. The tensile-modulus value of the WD/PF composite increased with the further addition of CP particles. The maximum modulus value was observed at 40 % mass fraction of the particles.

The results of the flexural tests of the WD/CP/PF composites with respect to the particle content are given in **Figure 2b**. It is interesting to note that the flexural strength and modulus of the WD/PF composite increase with the addition of CP particles. The flexural strength of the WD/PF composite is slightly lower than the value of the neat-resin sample. The maximum values of the flexural strength and modulus were identified at the 40 % addition. The flexural strength of the WD/PF composite

was increased by about 68.99 % due to the incorporation of 20 % mass fraction of CP particles.

The impact-strength values of the WD/CP/PF composites after the impact tests are presented in **Figure 2c**. It can be seen that the impact strength of the WD/PF composite is slightly lower than the value of the neatresin sample. It is also shown that the impact strength of the WD/PF composite increases with the addition of 10 % mass fraction of CP particles, but it decreases with the incorporation of 20 % mass fraction of CP particles. This may be due to a poor adhesion between the particles and the matrix. It may also be due to the stress concentration of the resin matrix. It is also observed that the incorporation of 10 % mass fraction and 20 % mass fraction of CP particles shows 15.57 % and 8.19 % higher impact values compared to the WD/PF composite.

3.2 Steady-state erosion: effects of the impingement angle

Generally, the erosive-wear behavior of polymer composite materials can be categorized as brittle and ductile. A ductile erosive situation is created in thermoplastic polymer composites, whereas the a brittle erosive situation may be created in thermosetting polymer composites. For the steady-state-erosion analysis of the WD/CP/PF composites, an erosion test was carried out based on eight different impingement angles (20, 30, 40,



Figure 2: Variation of: a) tensile property, b) flexural property, and c) impact strength based on the particle content



Figure 3: Steady-state erosive behavior of WD/CP/PF composites for eight different impingement angles

50, 60, 70, 80 and 90) $^{\circ}$, keeping all the other process parameters constant (the initial level values).

The effects of the impingement angles on the erosion rate of the WD/CP/PF composites are presented in Figure 3. From this figure, it can be observed that the erosion rate is high at the impingement angle of 60° for all the composite specimens, irrespective of the particle content. However, a more brittle erosive behavior was identified for the 40 % mass-fraction (20WD/20CP) composite specimen. However, in the 20 % and 30 % mass-fraction composite specimens, a semi-brittle erosive behavior was identified. This may be due to the addition of WD and CP particles to the PF composites. When the higher amounts of particles are added to the polymer material, it behaves as a typical brittle material. Therefore, the brittleness of the composites with 20 % mass fraction and 30 % mass fraction of particles is lower than the composite with 40 % mass fraction of particles. Due to this, a semi-brittle erosive situation exists during the erosive process. It is also clear from Figure 3 that the erosion rate increased with the increase in the particle content. This may be due to the increased hardness of the PF composite material caused by the addition of WD and CP particles.

3.3 Analysis of the erosion rate

The erosion rates for 27 combinations of the erosive experiments conducted on the WD/CP/PF composites are given in Table 3. The erosion analysis was made with popular software, namely, MINITAB 17. From Table 3, it can be concluded that the parameter combination of particle loading-A (level II = 30% mass fraction), impact velocity-B (level I = 41 m/s), impingement angle-C (level I = 30°), erodent size-D (level II = 500um) and standoff distance-E (160 mm) gives the minimum erosion rate (189. 8 mg/kg). Moreover, another parameter combination (experiment number 4) allows the next level of the minimum erosion rate (194.1 mg/kg). The difference between these two erosion rates is small, as seen from Table 3. Anyway, the first parameter combination mentioned above is recognized as the better combination of parameters to obtain the minimum erosion rate. The overall mean of the signal-to-noise ratio for the erosion rate is found to be 49.75 dB.



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Figure 4: a) Effects of erosive-process parameters and b) effects of interactions of erosive-process parameters on the erosion rate

 Table 3: The erosion rates and their S/N ratio of WD/CP/PF composites for 27 combinations

Experi			~		-	Erosion	S/N
Ment No.	А	В	С	D	E	rate mg/kg	ratio dB
1	20	41	30	300	80	209.5	-46.42
2	20	41	60	500	120	273.8	-48.75
3	20	41	90	700	160	220.7	-46.87
4	20	52	30	500	120	194.1	-45.76
5	20	52	60	700	160	267.3	-48.54
6	20	52	90	300	80	246.2	-47.82
7	20	63	30	700	160	211.9	-46.52
8	20	63	60	300	80	301.3	-49.58
9	20	63	90	500	120	277.1	-48.85
10	30	41	30	500	160	189.8	-45.65
11	30	41	60	700	80	343.5	-50.72
12	30	41	90	300	120	312.7	-49.90
13	30	52	30	700	80	298.9	-49.51
14	30	52	60	300	120	367.2	-51.29
15	30	52	90	500	160	351.3	-50.91
16	30	63	30	300	120	300.8	-49.56
17	30	63	60	500	160	378.5	-51.56
18	30	63	90	700	80	361.9	-51.17
19	40	41	30	700	120	332.8	-50.44
20	40	41	60	300	160	387.5	-51.76
21	40	41	90	500	80	370.6	-51.37
22	40	52	30	300	160	359.1	-51.10
23	40	52	60	500	80	398.3	-52.00
24	40	52	90	700	120	381.7	-51.63
25	40	63	30	500	80	379.2	-51.58
26	40	63	60	700	120	427.6	-52.62
27	40	63	90	300	160	369.8	-51.36

The effects of five erosive-process parameters on the erosion rate are graphically presented in **Figure 4a**. From this figure, it can be clearly concluded that parameter A (the particle content), parameter B (the impact velocity) and parameter C (the impingement angle) are the most significant parameters. Parameter E (the standoff distance) shows a moderately significant influence, while parameter D (the erodent size) has a relatively less significant influence. **Figure 4b** shows the interaction between the erosive parameters. From this figure, it is observed that a moderate interaction exists between parameters A and B, and between A and C. The interaction between parameters B and C is below the moderate level.

Figures 5a to **5c** show 3D surface plots of the erosion rate with significant process parameters. The observation is similar to the one made of the interaction plots of the erosion rate. From the erosion test analysis of the WD/CP/PF composites, it can be concluded that the erodent size is most insignificant for the erosion rate. The standoff distance shows relatively less significance when compared to the other three process parameters (particle content, impact velocity and impingement



Figure 5: 3D surface plots of erosion rate vs process parameters: a) $A \times B$, b) $A \times C$, and c) $B \times C$

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angle). It can be concluded that the combination of the parameters – the particle content at level II, the impact velocity at level I and the impingement angle at level I – gives the minimum erosion rate. Therefore, this combination is recognized as the best combination of the erosive-process parameters to get the minimum erosion rate within the selected parameter range.

3.4 Analysis of the variance for the erosion rate

Taguchi's analysis of variance can be used to find the set of significant parameters as well as their interactions in any system. In this study, ANOVA is used to understand the contribution of the parameters to the erosion rate and the effects of their interactions on the erosion rate of the WD/CP/PF composite. The ANOVA results for the erosion rate of the WD/CP/PF composite are given in **Table 4**. The last column (*p*-value) in this table indicates the highly significant parameters and their main effects depend upon the value of p. The p values for the particle content, impact velocity, impingement angle and standoff distance are 0.000, 0.023, 0.003 and 0.186, indicating their great influence on the erosion rate of the composite. The p values of the interactions of the process parameters show a moderate significance and a significance below the moderate level. Table 5 shows the responses for the S/N ratio (the small-the-better characteristic). The order of the erosive-process parameters based on their contributions to obtain the minimum erosion rate is the particle content, impingement angle, impact velocity, standoff distance and erodent size.

Table 4: Analysis c	of variance	for Means	of erosion rate
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Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	2	81404	81403.9	40701.9	120.99	0.000
В	2	7520	7519.9	3759.9	11.18	0.023
C	2	25188	25188.1	12594.0	37.44	0.003
D	2	97	96.7	48.4	0.14	0.870
E	2	1778	1778.4	889.2	2.64	0.186
A*B	4	2698	2698.2	674.6	2.01	0.258
A*C	4	3305	3305.3	826.3	2.46	0.203
B*C	4	791	791.1	197.8	0.59	0.690
Residual Error	4	1346	1345.6	336.4		
Total	26	124127				

Table 5: Response table for signal to noise ratios (smaller is better)

Level	А	В	С	D	Е
1	-47.68	-49.10	-48.51	-49.87	-50.02
2	-50.03	-49.84	-50.76	-49.61	-49.87
3	-51.54	-50.31	-49.99	-49.78	-49.37
Delta	3.86	1.21	2.25	0.26	0.65
Rank	1	3	2	5	4

3.5 Confirmation experiment

At the end of this study, we carried out a confirmatory experiment as the final test to validate the estimated results obtained during the erosive analysis of the WD/CP/PF composite. Therefore, the experimental results were verified with the estimated results using the confirmation test. This test was conducted to predict the erosion rate caused by a new set of erosive-process-parameter levels ($A_2B_1C_1E_3$). To predict the S/N ratio, the following equation can be used:

$$S/N_p = (A2-T) + (B1-T) + (C1-T) + (E3-T) + T$$
 (2)

where *T* is the overall experimental average of the *S/N* ratio and *S/N*_p is the value of the predicted S/N ratio. The comparison results for the predicted and experimental *S/N* ratio of the optimum process parameters are given in **Table 6**. The difference between the predicted and experimental *S/N* ratio is 0.51, i.e., an error of 1.1 %. It proves that the model can predict the erosion rate with a reasonable accuracy.

 Table 6: Comparison results of predicted and experimental signalto-noise ratio of optimal process parameters

	Optimal erosive process parameters				
	Predicted Experimental Differen				
Parameter level	$A_2B_1C_1E_3$	$A_2B_1C_1E_3$	Predicted- Experimental		
Erosion rate mg/kg	191.5	189.8	1.7		
Signal-to-noise ratio dB	-46.17	-45.66			

4 CONCLUSIONS

Mechanical properties of a WD/CP/PF composite were analyzed based on the particle content. The results show that the tensile strength of the WD/PF composite increased with the addition of 10 % mass fraction of CP particles, but decreased with the addition of 20 % mass fraction of CP particles. The flexural properties of the WD/PF composite increased with the increase in CP particles. The impact strength of the WD/PF composite also increased with the addition of 10 % mass fraction of CP particles and decreased with further addition of CP particles. The steady-state erosion analysis was carried out for eight different impingement angles on the WD/CP/PF composite. The composite with the lower particle content shows a semi-brittle erosive behavior with a higher erosion wear at the 60° impingement angle. On the other hand, the composite with the higher particle content showed a fully brittle nature of the erosive behavior with a higher erosion wear at the 60° impingement angle. From the erosive analysis of the WD/CP/PF composite, the process parameters like the particle content, impingement angle and impact velocity are found to be the most significant parameters influencing the erosion rate. The standoff distance shows a moderate influence on the erosion rate, while the erodent size shows a less significant influence on the erosion rate.

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